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Ion Transport Study in Corn Starch-NaHSO$_3$ Based Polymer Electrolytes

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ABSTRACT

In this work, different amounts of sodium bisulfite, NaHSO$_3$ (5wt.% to 40wt.%), were dissolved in a corn starch polymer host to synthesize solid polymer electrolyte (SPE) using a solution casting technique. Then, the films prepared were characterised using non-destructive electrical impedance spectroscopy to determine SPE films' conductivity and electrical transport properties. The highest conductivity of the sample at ambient temperature was recorded at 15wt. % of NaHSO$_3$ with a value of 2.22×10^{-4} Scm^{-1}. Moreover, the ion transport parameters at room temperature are found to be 2.41 × 10-7 cm^{2}s^{-1}, 9.39×10^{-6} cm^{2}V^{-1}s^{-1}, 1.70 × 1019 cm^{-3} for D, μ and n, respectively. The conductivity of the samples was found dependent on diffusion coefficient, D and mobility, μ of ions.

Keywords: impedance study, ion transport parameters, solid polymer electrolyte, conductivity

INTRODUCTION

Nowadays, technology development depends on improvement in solid state electrochemical devices such as batteries, fuel cells, sensors, and supercapacitors [1-5]. For these to be applicable at the industrial level, it needs proper electrolytes that contribute to net free ions transport. In this research, solid polymer electrolyte (SPE) was studied. SPE is widely used in rechargeable batteries and other electric devices compared to conventional liquid electrolytes. This is due to their attractive features, such as reducing corrosive solvent leakage, light in weight, flexible in shape for battery fabrication, and high mechanical strength [6-12]. Meanwhile, the liquid electrolyte has some drawbacks, which are leading to leakage and being difficult for storage as well as in packaging [13].

Currently, researchers are looking for an alternative to improve SPE properties from the usage of a suitable polymer host. Natural polymers such as starch, chitosan, and cellulose are widely used in solid polymer electrolytes as a host due to their characteristics which are renewable and environmental friendly [14, 15]. In this study, corn starch was selected as the polymer host material. It is biodegradable, low cost, abundant in nature, non-toxic, and has high solubility in water [16-20]. However, most of the natural polymers possess very low conductivity of thin films. Due to this reason, salt was added as a dopant in the electrolytes to enhance the conductivity.

Sodium-based electrolytes were chosen as an alternative to lithium ions. This is due to the properties of sodium (Na) salts suitable to be a dopant, such as low toxicity, natural abundance, lightweight, and high electrochemical potential [21-22]. Besides, Na also exhibits similar chemical properties to lithium, which can be applied in the battery system. Meanwhile, lithium raw materials are more expensive than Na, and it is also considered unsafe to use in the surrounding environment [23-26]. Hence, this study focuses on corn starch as a polymer host doped with sodium bisulfite as a dopant. As mentioned by Arof *et al.* (2014) [27], the non-destructive electrochemical impedance spectroscopy (EIS) technique was used in this study as a powerful tool in the determination of ionic conductivity and electrical transport properties for a wide range of electrolytes, including ionic SPE.

MATERIALS AND EXPERIMENTAL METHODS

Sample Preparation

Corn starch ($C_6H_{10}O_5$) and sodium bisulfite, $NaHSO_3$, with a purity of 96% was obtained from Sigma-Aldrich. Briefly, two different solvents (20 ml distilled water and 0.6 ml glycerine) with a purity of 100% and 96% were mixed. Then, 1 g of corn starch powder was dissolved into the prepared solution, and a various amount of $NaHSO_3$ (in weight percentage, wt. %) was added to the solutions as listed in Table 1. As mentioned by Awang *et al.* (2020) [28], the weight percentage was calculated using a formula as expressed in Equation 1;

$$wt.\% = \frac{x}{x+y} \times 100 \tag{1}$$

where x is the amount of salt in gram (g), y is the amount of corn starch (g), and weight percentage is the varying values in percentage for salt as an ionic dopant.

Table 1: Compositions of Corn Starch- $NaHSO_3$ SPE Film

SPE Samples	Ionic salt (wt. %)	Ionic salt (g)
A	0	0
B	5	0.053
C	10	0.111
D	15	0.177
E	20	0.250
F	25	0.333
G	30	0.429
H	35	0.538
I	40	0.667

The mixtures were stirred continuously with a magnetic stirrer until it turns into homogenous solutions at 60°C to 70°C. Next, the solution was cast into different plastic Petri dishes and left dry slowly at ambient temperature to form thin films. After that, the films were kept in desiccators (with silica gel) for a certain period to remove the water content. Figure 1 illustrated experimental steps in the schematic diagram and the preparation process known as the solution casting technique.

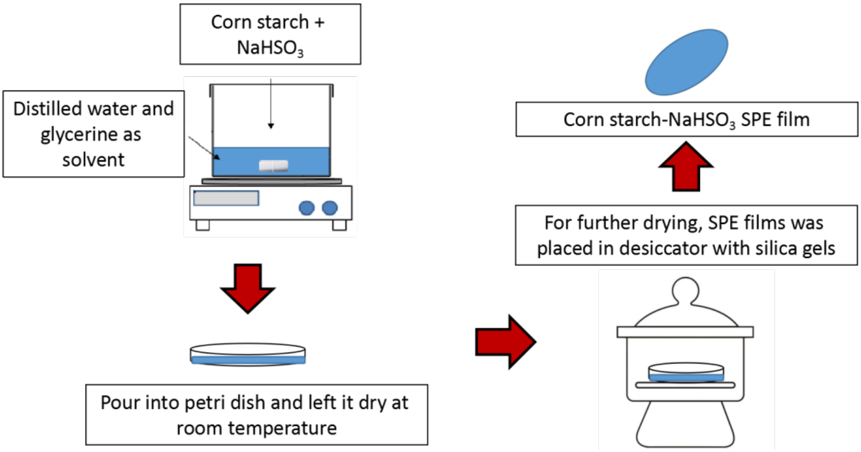

Figure 1: The Overall Process for SPE Preparation

EIS is a characterisation technique to determine the electrical properties of the studied SPE films. HIOKI 3532-50 LCR Hi-Tester, which was connected with a computer at room temperature, was used to measure the electrical impedance of the samples. The samples were cut with a dimension of 3.00 cm × 1.00 cm and sandwiched between two stainless steel blocking electrodes and being tested over the frequency range of 50 Hz to 1 MHz. Ionic conductivity of the SPE films was calculated by using Equation 2;

$$\sigma = \frac{l}{R_b A} \tag{2}$$

where σ is the ionic conductivity in (Scm^{-1}), l is the thickness of the samples (cm), A is the contact area of the blocking electrodes (cm^2), and R_b is the bulk resistance values in ohm obtained from the electrical impedance plot. The thickness of the films was measured by using a digital micrometre screw gauge, as shown in Figure 2.

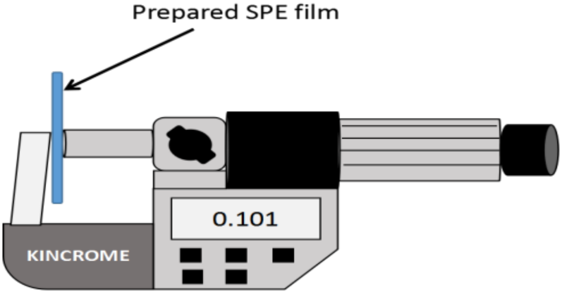

Figure 2: The Thickness of SPE Films are Measured

An examination of the conductivity behaviour of the SPE films is done using dielectric studies. This method could encourage researchers to comprehend the polarization impact at the interfaces of electrode-electrolyte. Also, the related formula was determined by Equations, as mentioned by Hassan and Azimi (2019) [29]. Dielectric constant (ε_r) for solid polymer electrolyte is defined by Equation 3.

$$\varepsilon_r = \frac{Z_i}{\left(Z_r^2 + Z_i^2\right)}\left(\frac{d}{\omega\varepsilon_0 A}\right) \tag{3}$$

From the equation, Z_r and Z_i are the real and imaginary part of complex permittivity (Ω), and d is the half-thickness of the polymer electrolyte (cm), ω is the angular frequency (corresponding to the minimum imaginary impedance or 2πf where f is a frequency in Hz), ε_0 is the permittivity of free space (8.85×10^{-14} Fcm^{-1}), and A is the contact area between electrode-electrolyte (cm^2).

In an attempt to calculate the transport properties of the electrolyte system, such as diffusion coefficient, mobility, and number density of charge carriers, the impedance study has been applied. In this work, the diffusion coefficient, D of ions charges, has been calculated using Equation 4 as shown.

$$D = \frac{\left(k_2\varepsilon_r\varepsilon_0 A\right)^2}{\tau_2} \tag{4}$$

Where k_2 is capacitance (F), ε_0 is the vacuum permittivity (F cm^{-1}), A is the contact area between electrolyte- electrode, cm^2 and τ_2 is a time constant (s) corresponding to the maximum dissipative loss curve, which is equal to $1/\omega_2$.

Another crucial parameter is ionic mobility, μ, which shows in Equation 5.

$$\mu = \frac{e\left(k_2 \varepsilon_r \varepsilon_0 A\right)^2}{k_b T \tau_2} \tag{5}$$

Where, k_b is the Boltzmann constant (1.38×10^{-23} J K^{-1}), T is the absolute temperature in Kelvin, and e is the electron charge (1.602×10^{-19} C). Then, the density number of mobile ions, n, which is in relation to conductivity, can obtain from Equation 6.

$$n = \frac{\sigma k_2 T \tau_2}{\left(e k_2 \varepsilon_r \varepsilon_0 A\right)^2} \tag{6}$$

Meanwhile, k_2 in Equation 4, 5, and 6 can be obtained from

$$k_2 = \frac{\lambda_D}{\varepsilon_r \varepsilon_0 A} \tag{7}$$

where, λ_D is the Debye length and its calculation obtained from Lin *et al.* [30].

$$\lambda_D = \sqrt{\frac{\varepsilon \varepsilon_0 k T}{Z^2 e^2 n}} \tag{8}$$

where Z = q is the mobile ion charge, e is the charge of an electron (Coulumb), n is the density of ion charge, ε is the relative dielectric permittivity (F m^{-1}), while ε_0 is the value of vacuum permittivity (8.854×10^{-12} F m^{-1}), T is the temperature in Kelvin.

RESULTS AND DISCUSSION

The solid polymer electrolyte (SPE) films were synthesized by using a casting technique. The selected images of SPE films with different content dopants as shown in Figure 3. As observed, the transparent corn-starch films were changed to translucent with an increasing amount of ionic salts. This happens because ionic dopants into the polymer electrolyte can alter the structure and morphology of the films [31]. Then, the thickness of the SPE samples varied between 0.101 mm to 0.293 mm.

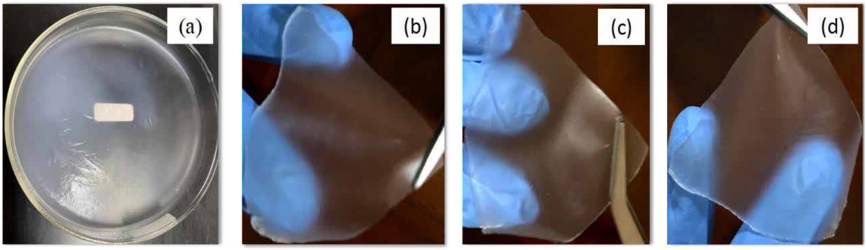

Figure 3: The Selected Pictures of SPE Films (a) Pure Corn Starch, (b) 5 wt. % of $NaHSO_3$, (c) 15 wt. % of $NaHSO_3$ and (d) 25 wt. % of $NaHSO_3$ (Source by Author)

Some examples of impedance plots of the polymer electrolytes were depicted in Figure 4. The bulk resistance (R_b) was determined by the intersection of the spike of a semicircle with the x-axis. From the figure, it can be said that the bulk resistance values were in contrast with the conductivity values. This can be seen as the sample that contains 15 wt. % of $NaHSO_3$ shows the lowest R_b values as tabulated in Table 2. At this level, the resistance at grain boundaries was suppressed due to higher mobile ions. So, it will increase the number of ion hopping which can contribute to the enhancement of conductivity.

Meanwhile, the sample with higher R_b values shows a declining trend of conductivity. This might happens because most of the immobile ions filled the bulk system. Hence, a smaller number of mobile ions would not contribute to the conductivity of the system.

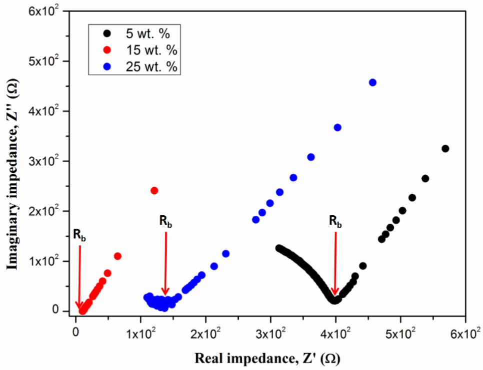

Figure 4: The Images of Bulk Resistance, R_b SPE Films Containing Different Amounts of $NaHSO_3$ (wt. %)

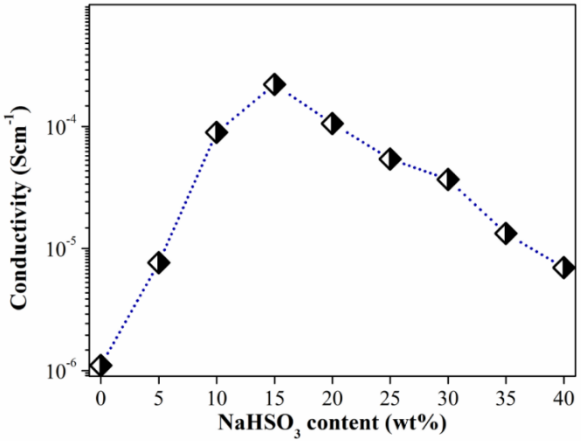

Figure 5: Effect of NaHSO$_3$ Content on the Conductivity of Electrolyte at Room Temperature

Figure 5 shows the variation of ionic conductivity for SPE samples. It can be said that the conductivity is due to the transport that can be related to ion-dissociation and aggregation formation. The ionic conductivity was achieved at 1.1 × 10^{-6} Scm^{-1} for 0 wt.% of NaHSO$_3$. It can be considered relatively high for an un-doped sample as corn starch contains high amylose content will have a highly amorphous phase [32]. The amorphous region is desirable that, in turn, will favor ionic mobility. Then, the addition of 5-15 wt. % NaHSO$_3$ into the polymer electrolyte influenced the increase of ionic conductivity SPE films until it reached the maximum value (2.22 × 10^{-4} Scm^{-1}). The increase in mobile ions and concentration of charged ions is affected by the increase in the amount of dopant. Thus, it will promote an increase in ionic conductivity [33]. Next, the conductivity started to decrease when 20 wt. % of NaHSO$_3$ was added with a value of 1.06 × 10^{-4} Scm^{-1}. A decrease in conductivity of SPE films after maximum conductivity has been achieved. This is also because the high concentration of salt can produce more dissociated ions which the distance between them becomes closer, and they can recombine and form neutral ion pairs [33]. Hence, this leads to a decrease in the density of mobile ions and a drop in the conductivity values. Besides, as mentioned by Barbosa *et al.* (2010) [35], it is expected that ionic conductivity increases with the rise in the amount of salts up to maximum and after that decreases. Addition of more NaHSO$_3$ salts of 25 wt. % to 40 wt. % eventually decreased the conductivity value until 6.98

$\times 10^{-6}$ Scm^{-1}, respectively. As a summarisation, the conductivity and bulk resistance data were mentioned in Table 2.

Table 2. Bulk Resistance and Ionic Conductivity of Corn Starch-NaHSO₃ SPE Films

SPE films (wt. %)	Bulk resistance, Rb (Ω)	Conductivity, σ (Scm^{-1})
0 (Pure corn starch)	3.00×10^{3}	1.10×10^{-6}
5	4.20×10^{2}	7.65×10^{-6}
10	6.00×10^{1}	8.96×10^{-5}
15	2.50×10^{1}	2.22×10^{-4}
20	4.00×10^{1}	1.06×10^{-4}
25	1.47×10^{2}	5.43×10^{-5}
30	1.60×10^{2}	3.69×10^{-5}
35	7.00×10^{2}	1.33×10^{-5}
40	1.17×10^{3}	6.98×10^{-6}

For experimental purposes, ion transport mechanisms of SPE films were obtained from the impedance results. The role of corn starch and $NaHSO_3$ is explored to understand the ion dynamics. The method was proposed by Arof, and his coworkers was used to get the values of dielectric constant, ε_r, for each sample [36]. This can be determined by a plot of the real part of complex permittivity, ε_r versus frequency, f that is shown in Figure 6 and substituted into Equation 3. It is observed that all the samples show a uniform value of the real part of complex permittivity, ε_r, in a range between log f = 5.5 and 6.0. Hence, the dielectric constant, ε_r values for all the samples were taken at 630 kHz. These dielectric constants were employed in Equations 4, 5, 6, 7, and 8. The obtained values λ_D, k_2, D, μ, and n are tabulated in Table 3. It was found that the λ_D had a value in the range between 1.25×10^{-5} and 7.74×10^{-7} cm, respectively. While the λ_D for the highest conductivity value was recorded at 2.77×10^{-6} cm. The k_2 value for the best conductivity sample was 4.37×105 F^{-1} in the range of 6.30×10^{6} and 1.09×10^{5} F^{-1}. The value of diffusion coefficient at the highest conductivity sample had achieved at 2.41×10^{-7} cm^{2} s^{-1}, and whereas the calculated value for all samples was between 2.05×10^{-6} and 6.77×10^{-7} cm^{2} s^{-1}, respectively. Moreover, the SPE films had mobility values in a range of 8.00×10^{-5} and 2.64×10^{-5} cm^{2} V^{-1}s with the value at the maximum conductivity was 9.39 $\times 10^{-6}$ cm$_2$ V^{-1}s. The value of the number density of charge carriers for corn starch- $NaHSO_3$ films was between 1.62×10^{18} and 1.28×10^{20} $cm^{-3,}$ with the value at the highest conductivity at 1.70×10^{19} cm^{-3}. From Table 3, it can be concluded that the diffusion coefficient and mobility of charge ions can influence the ionic conductivity value of the SPE films.

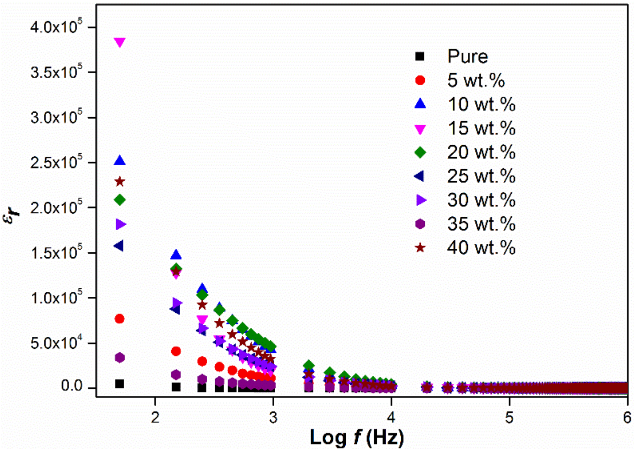

Figure 6: The Plot of Dielectric Constant (ε_r) Versus Frequency of SPE Films

Table 3: The Values of λ_D, k_2, τ_2, D, μ, and n of the Complex System

$NaHSO_3$ content (wt. %)	λ_D (cm)	k_2 (F^{-1})	τ_2 (s^{-1})	D ($cm^2\ s^{-1}$)	μ ($cm^2\ V^{-1}s$)	n (cm^{-3})
5	1.25×10^{-5}	6.30×10^{6}	2.65×10^{-5}	2.05×10^{-6}	8.00×10^{-5}	1.62×10^{18}
10	4.39×10^{-6}	6.92×10^{5}	1.22×10^{-6}	1.57×10^{-5}	6.13×10^{-4}	2.23×10^{17}
15	2.77×10^{-6}	4.37×10^{5}	3.18×10^{-5}	2.41×10^{-7}	9.39×10^{-6}	1.70×10^{19}
20	1.73×10^{-6}	3.47×10^{5}	2.65×10^{-6}	1.13×10^{-6}	4.39×10^{-5}	6.59×10^{18}
25	1.20×10^{-6}	2.82×10^{5}	5.30×10^{-6}	2.71×10^{-7}	1.06×10^{-5}	1.66×10^{20}
30	1.31×10^{-6}	1.57×10^{5}	3.18×10^{-6}	5.39×10^{-7}	2.10×10^{-5}	1.63×10^{20}
35	5.60×10^{-7}	2.31×10^{5}	3.94×10^{-5}	7.95×10^{-9}	3.10×10^{-7}	1.70×10^{22}
40	7.74×10^{-7}	1.09×10^{5}	8.84×10^{-7}	6.77×10^{-7}	2.64×10^{-5}	1.28×10^{20}

The values of D, μ, and n were plotted against the content of $NaHSO_3$ (wt. %) as depicted in Figure 7. At lower $NaHSO_3$ content, it was observed that n shows the same trend with conductivity and continues to oppose at higher $NaHSO_3$ content. Meanwhile, D and μ values are following the trend of conductivity. It seems that the D and μ of the charged ions may influence the trend of the ionic conductivity graph with the increase in $NaHSO_3$

concentration in SPE films. According to Hafiza and Isa (2017) [37], the trend of ionic conductivity depends on the adequate ionic conductors in the closed system and the amorphous structure of complex samples. Generally, the increase in ionic conductivity with increasing salt concentration can be related to the mobility and concentration of ions in polymer electrolyte films. However, the excess salt concentration will produce more free ions, which can lead to the ion aggregates and finally form a neutral ions pair. So, the mobility of charged ions will reduce. Thus, ionic conductivity also decreases. The addition of sodium salt in the polymer electrolyte also may increase the crystallinity of the membranes and result decreasing in ionic conductivity [38]. This might happens due to the interruption of intermolecular interactions within the host polymer to build a new interaction between molecules, thus influence the electrical properties of the SPE system [39]. Besides, the crystalline structure can also affect the mobility of the ions, limiting the ions to transport over the electrolyte system. Table 4 shows the comparison of conductivity and transport properties values of this study and other systems. It can be observed that the results studied here showed for corn starch-$NaHSO_3$ SPE films had best conductivity (2.22×10^{-4} Scm^{-1}) with transport properties ($D = 2.41 \times 10^{-7}$ cm^2s^{-1}, $\mu = 9.39 \times 10^{-6}$ $cm^2V^{-1}s^{-1}$ and $n = 1.70 \times 10^{19}$ cm^{-3}) to those studies in similar based starch systems. From that, it can be said the diffusion coefficient and mobility of charge ions may influence the conductivity values, and these results are vital as references in future works.

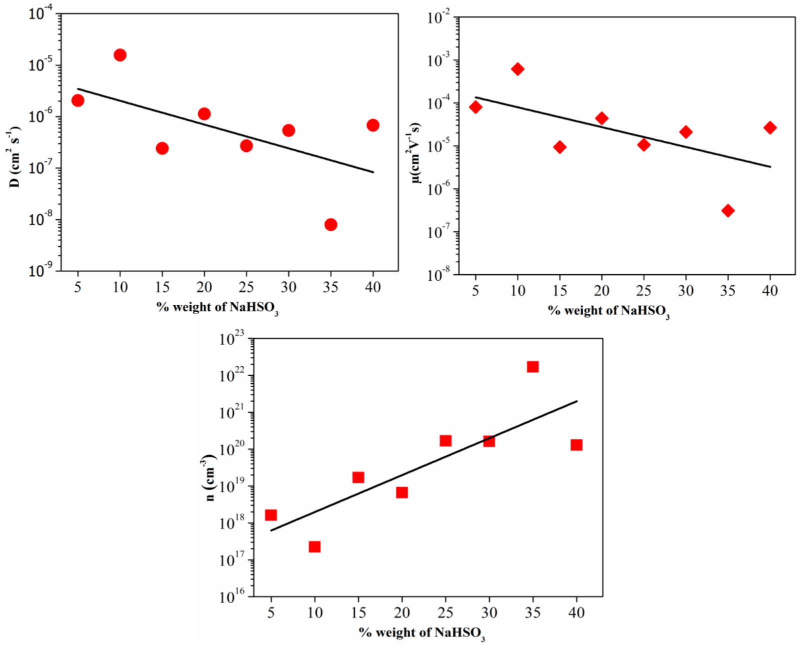

Figure 7: The Trend of Diffusion Coefficient, D, Mobility of Ion Charges, μ and Number Density of Charge Carriers, n Against $NaHSO_3$ Concentration

Table 4: The Comparison Values of Conductivity and Transport Properties (D, μ, and n) of This Work and Other Systems

System	Transport parameters			Conductivity (Scm^{-1})	References
	D ($cm^2\ s^{-1}$)	μ ($cm^2\ V^{-1}s$)	n (cm^{-3})		
Corn starch-$NaHSO_3$	2.41×10^{-7}	9.39×10^{-6}	1.70×10^{19}	2.22×10^{-4}	In this work
Corn starch-$MgSO_4$	1.04×10^{-8}	4.03×10^{-7}	1.31×10^{22}	8.52×10^{-5}	[40]
PEO-PVP-$NaNO_3$	1.87×10^{-4}	6.93×10^{-5}	2.63×10^{16}	2.92×10^{-5}	[41]
PVA-$LiCF_3SO_3$	9×10^{-8}	3×10^{-6}	0.8×10^{17}	-	[42]
CA-$NH4NO_3$	1.40×10^{-7}	5.30×10^{-6}	3.00×10^{19}	2.53×10^{-5}	[43]

Hexanoyl chitosan-$LiCF_3SO_3$-DEC/EC	-	1.41×10^{-5}	1.27×10^{18}	2.86×10^{-6}	[44]
Starch-chitosan-NH_4I	-	3.45×10^{-4}	5.50×10^{18}	$(3.04\pm0.32)\times10^{-4}$	[45]
Corn starch-NH_4Br	-	-	-	$(5.57\pm1.88)\times10^{-5}$	[46]

CONCLUSION

A solid polymer electrolyte-based corn starch has been developed by adding varying concentrations of $NaHSO_3$ via a solution casting technique. The ionic conductivity and transport parameters of SPE films were characterised by the EIS method. The decrease in diffusion coefficient and ionic mobility are results in the decrease of conductivity due to higher $NaHSO_3$ concentration. The SPE films exhibit the best conductivity 2.22×10^{-4} Scm^{-1} at ambient temperature. This enhancement in conductivity was contributed by the proper salt dissociation. The role of polymer and salt in the electrolyte system has been highlighted in ion transport mechanisms. It may be concluded that the present SPE film is suitable for use as an electrolyte (separator) for application in the battery system.

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