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Employing an Electrochemical Impedance Spectroscopy Technique to Estimate the Ion Transport Parameters in Corn Starch Based Solid Polymer Electrolyte

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Abstract: *The present work discusses the ionic conductivity and transport properties of solid polymer electrolyte (SPE). The SPE incorporates corn starch and sodium iodate (NaIO3) in various weight percentages prepared by a solution casting technique. The ionic conductivity, diffusion coefficient (D), ionic mobility (µ) and number on mobile ions (n) of SPEs were characterized by using an electrochemical impedance spectroscopy (EIS). From EIS, the highest ionic conductivity at room temperature was found to be 1.08 x 10-4 Scm-1 for 3 wt. % of NaIO3 and it is found that the ionic conductivity is dependent on the diffusion coefficient and mobility of freely charge ions.*

Keywords: Corn starch, sodium iodate, ionic conductivity, transport properties

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1. Introduction

Nowadays, the huge production of battery has created many environmental problems which can bring the negative effect to the surrounding as well as human health. It is urge to the researcher for thinking an alternative to overcome this issue. In order to reduce the related problems, natural based product has been chosen to be used in the electrolyte as one of the crucial battery components. For these to be applicable in a large scale, it needs for proper electrolytes which can be seen in solid polymer electrolyte (SPE).

2. Literature review

An electrolyte behaves as a conductive medium that concentrated with free ions. It can be categorized into liquid and solid electrolytes. According to [8-10, 31], there are a few disadvantages of liquid electrolytes such as poor mechanical properties, leakage of solvents and difficulty for storage which limit its usefulness. These drawbacks are believed could be overcame by introduction of solid polymer electrolyte (SPE). The usage of SPE in some electronic devices such as rechargeable batteries, fuel cells, sensors, supercapacitors and solar cells [1-4] were driven by their unique features and capability. For example, it is enable to reduce corrosion leakage, lighter, flexible in shape which easy to fabricate and good mechanical properties [5-7]. Generally, SPE is composed of ionic salts that dissolved in a suitable host polymer. The selection of natural polymers as a host likes starch [11], cellulose [12, 14], chitosan [13] and pectin are appeared to be a noble option. Among these well-known natural polysaccharides, corn starch based polymer electrolytes are found to be one of the most promising materials for Na-ion batteries [33].

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Numerous works have been extensively studied on biodegradable, low cost and renewable polymer materials to prepare SPE with high ionic conductivity and environmental friendly. However, only a few studies based on starch-solid polymer electrolyte had been reported, such as starch- NaSCN [15], starch- NH_4NO_3 [16], starch-NH_4I [17], starch- LiTFSI [11] and starch-$LiPF_6$ [18]. Most of the conductivity of the mentioned systems are found to be less than 10^{-4} Scm^{-1} with no conduction mechanism had been discussed in their systems which is a crucial part to genuinely understand the characteristic of the studied SPE. Dealing with ionic conductivity of polymer electrolytes is vital in order to alter the structure of the materials which can improve its chemical and physical properties. Therefore, three basic factors that can influence the conductivity in SPE such as diffusion coefficient, concentration and mobility of ions. Besides, common concept to estimate these transport parameters are using an electrical impedance spectroscopy (EIS), Bandara and Mellandar approach and FTIR technique.

In this research, ion transport parameters for corn starch- $NaIO_3$ SPE has been investigated using a non-destructive EIS technique as mentioned by [32]. From our knowledge, there is no report on the preparation and characterization of SPE doped with sodium iodate ($NaIO_3$). Hence, the main purpose of the present work is to study the effect of $NaIO_3$ salt concentration on conductivity and transport properties of starch based solid polymer electrolyte using a new calculation technique which is rarely reported before.

3. Materials and Experimental methods

Sample preparation

SPE films were prepared using a solution casting technique, added with various ratios of sodium iodate ($NaIO_3$) from Sigma-Aldrich in a range between 0-9 weight percentage (wt.%) of salt content as listed in Table 1. The weight percentage was calculated using a formula as expressed in Eq. 1;

$$wt.\% = \frac{x}{x+y} \times 100 \qquad (1)$$

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

Corn starch obtained from Sigma-Aldrich with linear formula $C_6H_{10}O_5$ was dissolved in two different solvents (20 ml distilled water and 0.6 ml glycerine) with a purity of 100% and 96% and mixed with previous weighted $NaIO_3$. The mixtures were stirred continuously until it turns to a homogenous solution. Then, the solution was poured into different petri dishes and left it dry naturally at ambient temperature to form thin films. For further drying process, SPE thin films were kept in a desiccator filled with silica gels to eliminate moisture. The average thickness of SPE films was 0.101 mm to 0.293 mm measured using a digital micrometer screw gauge (Kincrome 5610). This experimental process was summarized in Figure 1.

Table 1: Ratios of corn starch SPE films.

Samples	Sodium iodate (wt.%)	Sodium iodate (g)	Corn starch (g)
Pure	0	0	1
A	1	0.010	1
B	2	0.020	1
C	3	0.031	1
D	4	0.042	1
E	5	0.053	1

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F	6	0.064	1
G	7	0.075	1
H	8	0.086	1
I	9	0.097	1

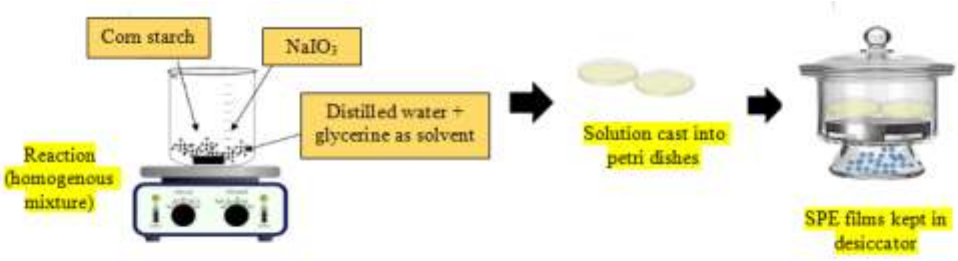

Figure 1: Schematic diagram of SPE preparation process.

Characterization

Conduction mechanism properties of SPE films were characterized using electrochemical impedance spectroscopy (EIS) analysis. All the samples were tested at room temperature using HIOKI 3532-50 LCR Hi-Tester which interfaced with a computer at a frequency range of 50 Hz to 1MHz. The samples were cut into dimensions (0.03 m x 0.01m) and sandwiched between two stainless steel blocking electrodes with a diameter of 2.2×10^{-2} m.

The values of bulk resistance, R_b for each sample was obtained from the Nyquist plot and conductivity was calculated using Equation 2.

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

Where l is the thickness of the SPE films and A is the surface area contact between electrode-electrolyte (cm^2) with a value of 3.142 cm^2.

The best way to describe the conductivity behaviour of SPE by using dielectric studies. Dielectric constant, ε_r for each sample can be determined from a plot of the real part of complex permittivity, ε_r versus frequency, f and substituted into Equation 3. All the samples are investigated in a range between log f = 5.5 and 6.0. Hence, the dielectric constant, ε_r values were taken at 630 kHz.

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

where Z_r and Z_i are the real and imaginary part of complex permittivity and d is the half thickness of SPE films, ω is the angular frequency ($\omega = 2\pi f$ where f is a frequency in Hz), ε_0 is the permittivity of free space (8.85×10^{-14} F cm^{-1}) and A is the area of blocking electrode contact.

The parameter's value such as diffusion coefficient (D), mobility of charge carriers (μ) and number density of charge carriers (n) can be calculated using Equation 4, 5 and 6, respectively.

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

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

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$$n = \frac{\sigma k_2 T\tau_2}{(ek_2\varepsilon_r\varepsilon_r A)^2} \qquad (6)$$

According to Equation above, k_2 is capacitance, ε_0 is the vacuum permittivity, A is the surface contact between electrolyte- electrode and τ_2 is a time constant corresponding to the maximum dissipative loss curve which is equal to $1/\omega_2$, k_b is the Boltzmann constant (1.38 x 10^{-23} J K^{-1}), T is the absolute temperature in Kelvin and e is the electron charge (1.602 x 10^{-19} C). Then, k_2 can be determined from equation 7,

$$k_{2}= \frac{\lambda_D}{\varepsilon_r\varepsilon_0 A} \qquad (7)$$

As reported by [20], the values of Debye length, λ_D can be calculated from Lin et al., (2012) as shown in equation 8.

$$\lambda_D = \sqrt{\frac{\varepsilon\varepsilon_0 kT}{Z^2e^2n}} \qquad (8)$$

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

4. Results and Discussion

Figure 2: Corn starch-$NaIO_3$ films

Figure 2 shows the images of corn starch-$NaIO_3$ SPE film at room temperature. After dissolution of $NaIO_3$ into electrolyte, the SPE had transform from white to the dark purple color. Then, the SPE was left dry naturally until it forms a thin film. The dried SPE shifted the color into a pale purple and it took 24 hours for the SPE film to finally turn colourless.

The complex impedance is a technique applied at various frequencies to investigate the mechanisms of ions transport and charged transfer in both electrode and electrolyte [36]. It is plotted to determine the bulk resistance, R_b values. Basically, it consists of a title spiked, a depressed semicircle or a combination of a depressed semicircle with title spiked [35]. Figure 3 shows a cole- cole plot for selected SPE of corn starch doped with different concentrations of $NaIO_3$. From the plot, the values of R_b was determined at the intersection between the spiked and semicircle.

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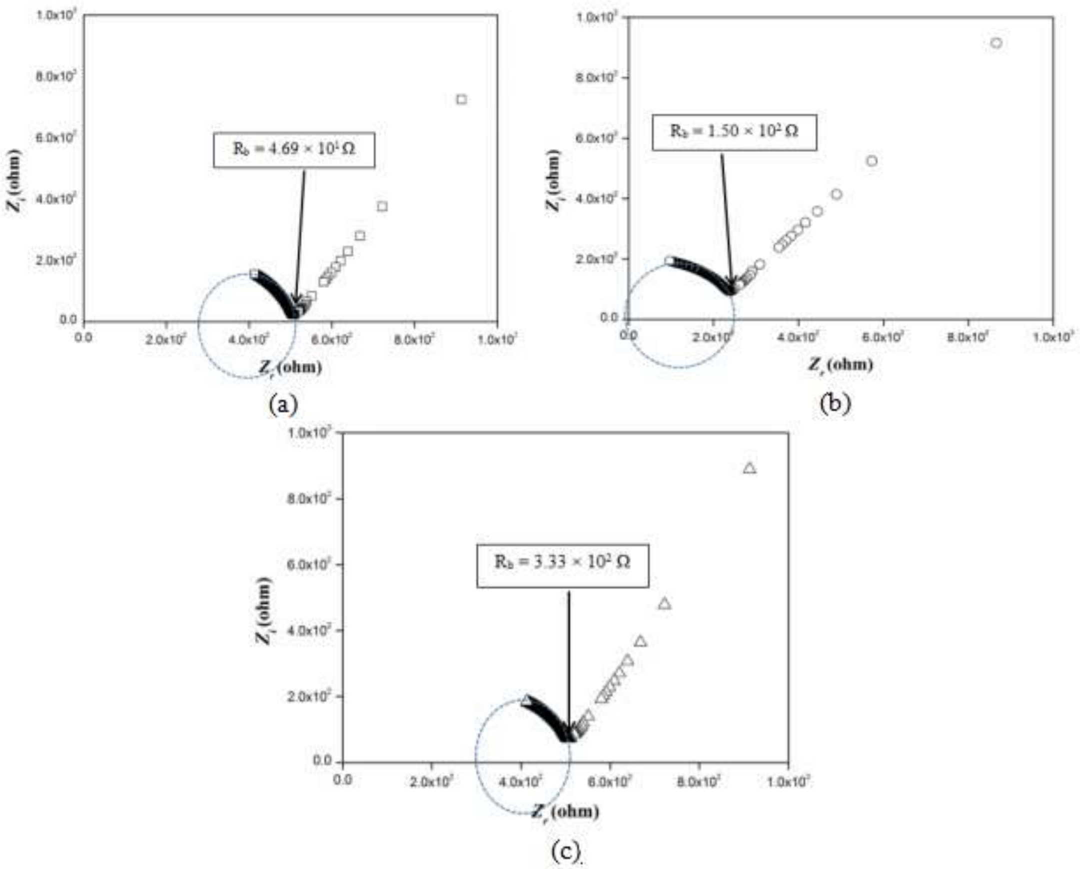

Figure 3: The impedance plot at ambient temperature of different concentration $NaIO_3$ (a) 3 wt.% of $NaIO_3$ (b) 5 wt.% of $NaIO_3$ and (c) 7 wt.% of $NaIO_3$.

The ionic conductivity of corn starch-$NaIO_3$ is shown in Figure 4 while the conductivity and bulk resistance values are tabulated in Table 2. It can be observed that the ionic conductivity of pure corn starch at room temperature is 1.1×10^{-6} Scm^{-1}. Then, for SPE films that contained 1 and 2 wt.% of $NaIO_3$, ionic conductivity increases to 2.15×10^{-5} Scm^{-1} and 3.43 $\times 10^{-5}$ Scm^{-1}. The increase in conductivity is due to the decreasing value of R_b. Upon addition of 3wt.% of $NaIO_3$, the highest conductivity value is achieved at 1.08×10^{-4} Scm^{-1}. The conductivity starts to decrease when there is an addition of 4 wt.% $NaIO_3$ with a value of 7.03 $\times$ 10^{-5} Scm^{-1}. Additional content of $NaIO_3$ in SPE continues to decrease the conductivity values until 7.9×10^{-6} Scm^{-1}. Conductivity values seem to decrease after it reaches the highest point. This behaviour may be affected by overcrowding of ions which can attribute to the formation of ions aggregates [21, 25]. Also, the bigger size of contact ions forms will limit the movement of ions in SPE. So, free mobile ions will decrease as well as reduce the number of density free mobile ions, hence decreasing the conductivity [22, 23]. In general, it can be said that the ionic conductivity of SPE is related to the number and mobility of ions.

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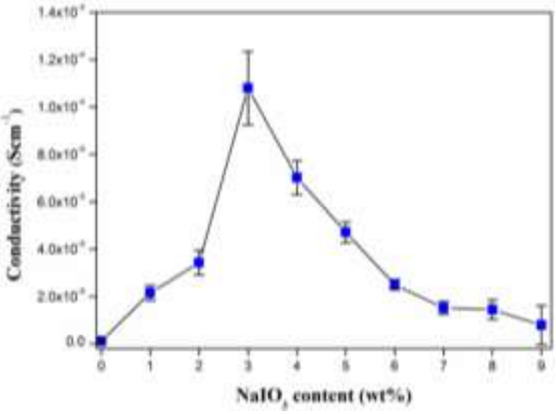

Figure 4: Ionic conductivity of corn starch- NaIO$_3$ films.

Table 2: The bulk resistance and conductivity of each samples.

Samples	**Bulk resistance, R_b (Ω)**	**Conductivity, σ (Scm^{-1})**
Pure	3.00×10^3	1.10×10^{-6}
A	2.00×10^2	2.15×10^{-5}
B	1.80×10^2	3.43×10^{-5}
C	4.69×10^1	1.08×10^{-4}
D	6.20×10^1	7.03×10^{-5}
E	1.50×10^2	4.71×10^{-5}
F	2.33×10^2	2.50×10^{-5}
G	3.33×10^2	1.52×10^{-5}
H	4.66×10^2	1.44×10^{-5}
I	5.00×10^2	7.90×10^{-6}

The transport parameters play an important role to explain the charge transfer and ionic transport process in materials. The fundamental of the parameters is diffusion coefficient (D), mobility of the ions (μ) and number density of ions (n). All the transport properties that are related to the ionic conductivity of SPE are recorded in Table 3, while the graph of D, μ and n data against NaIO$_3$ content are plotted using the Origin software as shown in Figure 5. Generally, according to [29], the ionic conductivity in SPE is related to the number and mobility of ions. However, in this study, diffusion coefficient and mobility give a more significant contribution to the conductivity of SPE films compared with number density.

This can be seen for diffusion coefficient values, the highest conductivity film had a value around 1.03×10^{-7} cm^2 s^{-1} and whereas, the measured value for all SPE samples was in between 3.12×10^{-6} and 5.73×10^{-7} cm^2 s^{-1}, respectively. Furthermore, the mobility values of ion charges in a range of 1.22×10^{-4} and 2.23×10^{-5} cm^2 V^{-1}s, and 4.01×10^{-6} cm^2 V^{-1}s for the highest conductivity. The value of the number density of ion charges for corn starch- NaIO$_3$ samples was between 1.06×10^{18} and 1.12×10^{20} cm^{-3} with the highest conductivity at 3.98×10^{19} cm^{-3}.

In general, the dielectric constant indicates the number of charged ions that can be stored by a material. It is also can be used as an indicator to prove that the increase in conductivity is due to the increase in free mobile ions [24]. According to [37], when the composition of salt increases, the number density of free ions will increase too. So, the charge stored in material also increases. From the result, it can be observed that the D and μ had followed the conductivity trend meanwhile the n shows the opposite trend. This is because the higher rate of diffusion and mobility of charge carriers in SPE can influence the increase of conductivity [30]. From literature, it may be concluded that the movement of ions is influenced by the

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addition of salt. Table 4 shows the comparison value of transport properties in this research with other systems.

Table 3: The values of λ_D, k_2, τ_2, D, μ and n of the corn starch-$NaIO_3$ films.

$NaIO_3$ 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})
1	9.10×10^{-6}	1.47×10^{6}	2.65×10^{-5}	3.12×10^{-6}	1.22 x 10^{-4}	1.06 x 10^{18}
2	3.74×10^{-6}	8.15×10^{5}	1.22×10^{-6}	1.14×10^{-5}	4.45 x 10^{-4}	3.07 x 10^{17}
3	1.81×10^{-6}	6.59×10^{5}	3.18×10^{-5}	1.03×10^{-7}	4.01 x 10^{-6}	3.98 x 10^{19}
4	2.08×10^{-6}	2.88×10^{5}	2.65×10^{-6}	1.63×10^{-6}	6.35 x 10^{-5}	4.56 x 10^{18}
5	1.38×10^{-6}	2.43×10^{5}	5.30×10^{-6}	3.59×10^{-7}	1.40 x 10^{-5}	1.26 x 10^{20}
6	9.19×10^{-7}	2.22×10^{5}	3.18×10^{-6}	2.65×10^{-7}	1.03 x 10^{-5}	3.10 x 10^{20}
7	7.00×10^{-7}	1.80×10^{5}	3.94×10^{-5}	1.24×10^{-8}	4.84 x 10^{-7}	1.09 x 10^{22}
8	5.35×10^{-7}	1.58×10^{5}	8.84×10^{-7}	3.24×10^{-7}	1.26×10^{-5}	2.69 x 10^{20}
9	5.16×10^{-7}	1.04×10^{5}	4.65×10^{-7}	5.73×10^{-7}	2.23×10^{-5}	1.12 x 10^{20}

Table 4: Comparison values of conductivity and transport parameters of this work and other systems.

Systems	Conductivity (S cm^{-1})	Transport parameters			References
		D ($cm^2 s^{-1}$)	μ ($cm^2 V^{-1}s$)	n (cm^{-3})	
Corn starch-$NaIO_3$	1.08×10^{-4}	1.03×10^{-7}	4.01 x 10^{-6}	3.98 x 10^{19}	This present work
MC- NH_4I	5.08×10^{-4}	6.15×10^{-6}	2.39×10^{-2}	1.32×10^{19}	[26]
MC-NH_4NO_3	2.10×10^{-6}	-	2.70×10^{-6}	4.86×10^{18}	[27]
CMC-NH_4SCN	6.48×10^{-5}	8.60×10^{-11}	3.29×10^{29}	1.23×10^{23}	[28]
Chitosan acetate-adipic acid	1.40×10^{-9}	6.65×10^{-11}	2.59×10^{-9}	3.38×10^{18}	[29]
Chitosan acetate- NH_4NO_3	2.53×10^{-5}	1.40×10^{-7}	5.30×10^{-6}	3.00×10^{19}	[30]
MC-AF	6.40×10^{-7}	1.19×10^{-11}	4.64×10^{-10}	-	[31]

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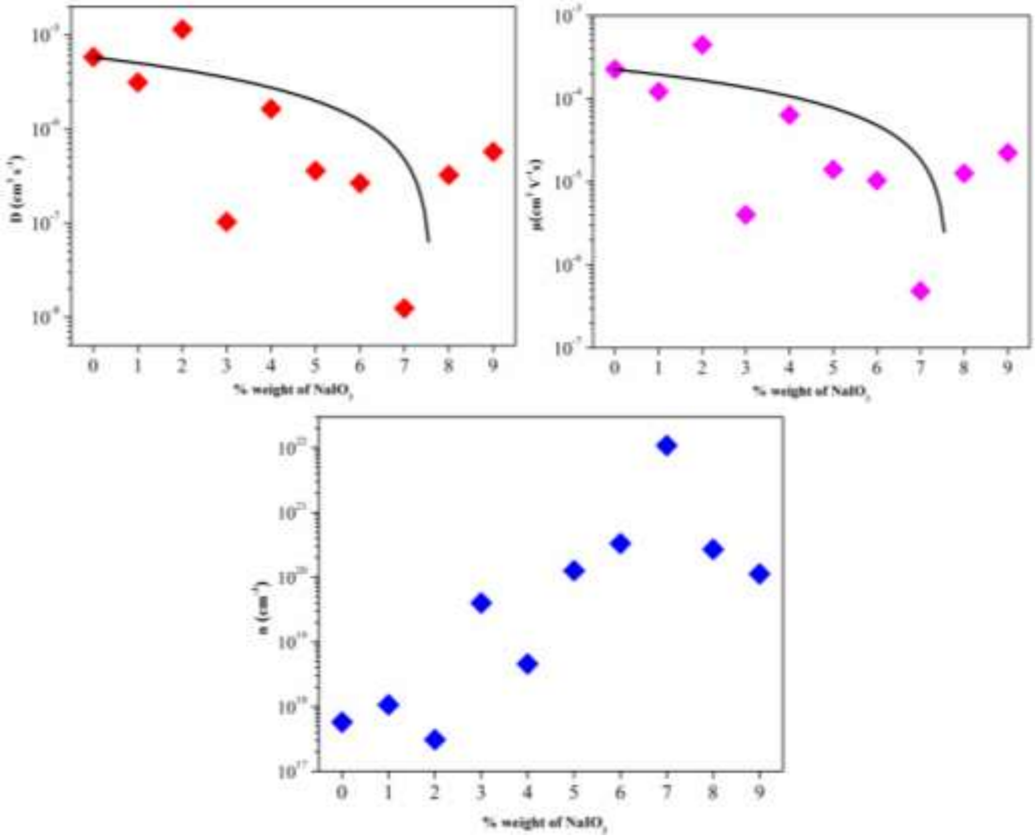

Figure 5: The plot of diffusion coefficient, *D*, mobility of ion charges, *μ* and number density of charge carriers, *n* versus NaIO$_3$ content.

5. Conclusion

Corn starch-NaIO$_3$ solid polymer electrolyte has been prepared using a solution casting technique. The ionic conductivity and conduction mechanisms (*D*, *μ* and *n*) of SPE films were characterized by electrical impedance spectroscopy (EIS) analysis. The ionic conductivity of SPE films increases to the optimum value of 1.08 x 10^{-4} Scm^{-1} with the addition of NaIO$_3$ content. The transport properties of SPE films such as mobility, number of mobile ions and diffusion coefficient have been calculated using the EIS method. The decrease in diffusion coefficient and ionic mobility results in the decrease of conductivity due to higher NaIO$_3$ concentration. It may be concluded that the prepared SPE films have the potential to be used as an electrolyte for application in energy storage devices.

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