

CORN STARCH DOPED WITH SODIUM IODATE AS SOLID POLYMER ELECTROLYTES FOR ENERGY STORAGE APPLICATIONS

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ABSTRACT. The concern about environmental problems has inspired a of energy storage devices from natural sources. In this study, solid polymer electrolyte (SPE) films made from corn starch doped with different compositions of sodium iodate (NaIO_3) were prepared via the solution casting technique. The effect of dopants on the structure, morphology and electrical properties of SPE films was analysed using X-Ray diffraction (XRD), scanning electron microscopy (SEM) and electrochemical impedance spectroscopy (EIS) analysis. From the XRD, it shows that the amorphous state would influence the conductivity values of SPE films. Then, the SEM observations revealed that the films seem to be rough, porous and having branch structure, which may affect the conductivity of SPE films. The maximum conductivity of SPE film is obtained from 3 wt.% of NaIO_3 with a value of $1.08 \times 10^{-4} \text{ Scm}^{-1}$ at room temperature (303K). From the results, this SPE is proposed to have a great potential in future energy storage applications.

KEYWORDS: Biodegradable polymers, polymer membranes, solid polymer electrolytes, sodium salt, electrical properties.

1. INTRODUCTION

Electrolyte is one of the main components in battery, which has been used as a medium for ion movement between anode and cathode. It can be divided into liquid and solid electrolytes. According to [1], there are a few limitations of liquid electrolytes, such as leakage issues and the difficulty to store and produce them. In order to solve the arising problems, solid polymer electrolyte (SPE) has been considered. The important criteria in the selection of SPE include stability and safety, reduction in cost and weight, and flexibility, which is easy to be fabricated and could perform as a proper medium for electrode-electrolyte contact [2–6]. Furthermore, it is seen suitable to be applied in some applications of electronic devices such as batteries, solar cell, sensors and supercapacitors [7–11]. However, the main barrier that makes SPE hard to be utilised is the relatively low ionic conductivity at ambient temperature. Normally, the ion conduction usually takes place in the amorphous region [12]. This is because the amorphous phase provides free spaces for the ions to move along with the polymer matrix. Many efforts have been suggested to enhance the ionic conductivity of polymer electrolytes, which includes incorporating various ionic dopant with host polymers [13].

The usage of biodegradable polymers is vital as they can be decomposed by a natural agent easily. Besides, they also take a short duration to decay as compared to non-biodegradable ones without leaving

any electronic waste. Some examples of polymers, such as chitosan, starch, pectin and cellulose [14–16] are usually used in certain areas. It is commonly used as a host for the preparation of solid polymer electrolyte (SPE) due to its properties, such as being renewable, cheaper, non-toxic and soluble in water [17–20]. In the SPE preparation, starches are used as host polymers and sodium iodate (NaIO_3) as an ionic dopant. Basically, starches are carbohydrate polymers that consist of amylose and amylopectin, which are bonded together [21]. However, the starch chains are held together by hydrogen bond, making them insoluble in cold water. Then, to obtain corn starch thin films, starch granules should be incorporated with a plasticizer in excess water at temperatures below 100°C . Examples of commonly used plasticizers are glucose, glycerol and sorbitol [22]. According to [23], glycerol is the most suitable plasticizer because of its mechanical characteristics and transparency. Also, the increase in glycerol contents can promote mobile ions to penetrate the polymer matrix. From this, it will facilitate the rise in amorphicity of the electrolyte and thus, leads to an increase in conductivity [24].

Salts act as an important constituent to an electrolyte as they have a strong influence on the electrolyte properties such as conductivity and thermal stability. In this study, sodium (Na) salts were selected as a well-established candidate due to their material abundance, low cost and environmentally friendliness [25–27]. Moreover, sodium also exhibits similar chemical properties to lithium that are applied

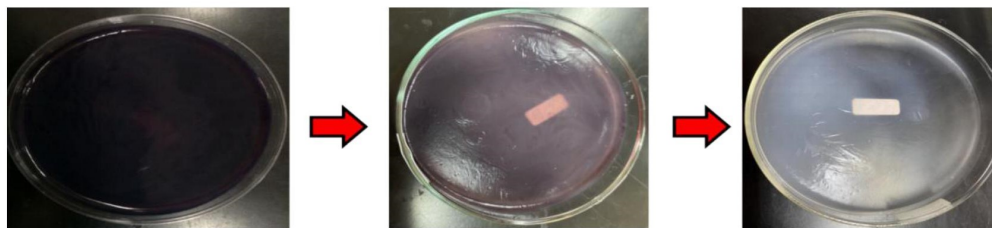


FIGURE 1. The physical changes of the SPE films.

in battery applications. Meanwhile, lithium (Li) materials themselves are very hazardous and the sources are very limited. Thus, the purpose of this study is to investigate the morphology and electrical properties of corn starch doped with sodium iodate (NaIO_3) by using the X-ray diffraction (XRD), scanning electron microscopy (SEM) and electrical impedance spectroscopy (EIS) techniques.

2. EXPERIMENTAL

2.1. MATERIALS

The solution casting technique was used to prepare corn-starch-based polymer electrolyte. Sodium iodate, NaIO_3 , (purity 96 %, Sigma-Aldrich) and corn starch, $\text{C}_6\text{H}_{10}\text{O}_5$, (Sigma-Aldrich) were used as the raw materials. In addition, two different solvents (20 ml of distilled water and 0.6 ml of glycerine) with a purity of 100 % and 96 % were also used.

2.2. PREPARATION OF SPE FILMS

SPE was prepared via the solution casting method. Different amounts of NaIO_3 (wt. %), which range from 0, 1, 3, 5, 7 and 9, were dissolved in the mixed solvents (distilled water and glycerine) until a complete dissolution. The weight percentage was calculated using the formula expressed in Equation 1.

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

Where, x is the weight of salt (g), y is the weight of the polymer and weight percentage is the difference values in percentage for salt as a dopant.

Thereafter, 1 g of corn starch was added into the solution and continuously stirred at 60-70 °C, until it turned homogenous with no phase separation. The solution was poured into glass petri dishes and left at room temperature for the SPE to be formed. For further drying process, the SPE films were kept in a desiccator filled with silica gels to eliminate any excess moisture.

2.3. CHARACTERISATION TECHNIQUE

X-ray diffraction (XRD) is a technique to determine the structural changes in a polymer electrolyte system. The crystallinity or amorphousness of the polymer electrolyte systems was observed using a MiniFlex II

diffractometer with $\text{CuK}\alpha$ radiation. The 2θ angle varies from 10° to 60° with a step size of 5 °/min per SPE film samples. Then, the obtained data were analysed using the Search-Match and Origin software. The degree of crystallinity (χ_c) of the SPE films was calculated from Equation 2.

$$\text{Crystallinity (\%)} = \frac{A_c}{A_c + A_m} \times 100 \quad (2)$$

Where, A_c and A_m are the crystalline and amorphous areas, respectively.

Scanning electron microscopy (SEM) was used for a morphological investigation. For this purpose, SPE films were coated to make them conductive for a better observation and placed on specimen tubs with double cellophane. They were examined using the Model JEOL JSM-6350LA with an acceleration voltage of 20kV at room temperature. The images were taken at various magnifications of $\times 350$, $\times 750$ and $\times 1000$. Through this method, the structure of the SPE films can be directly observed.

The electrical properties of prepared films were observed by the electrochemical impedance spectroscopy (EIS) technique. The thickness of the SPE films was measured using a digital micrometre screw gauge. Furthermore, the impedance of SPE films was determined using a HIOKI 3532-50 LCR Hi-Tester at room temperature, which is interfaced to a computer over a frequency range of 50 Hz to 1 MHz. The films were cut into appropriate size (3.0 cm \times 1.0 cm) and placed between two stainless blocking electrodes. The values of bulk resistance, R_b obtained from the measurement were used to calculate the conductivity, σ using Equation 3, respectively.

$$\sigma = \frac{l}{R_b A} \quad (3)$$

Where, l is the thickness of SPE films, R_b is the bulk resistance achieved from the interception of imaginary impedance against real impedance and A is the contact area of electrode-electrolyte.

3. RESULTS AND DISCUSSION

The physical image of the corn starch- NaIO_3 SPE films can be seen in Figure 1. It was observed that an insertion of NaIO_3 salt into the polymer electrolyte

had changed the homogenous solution into dark purple after being cooled at room temperature. Then, the SPE films were left to be dried naturally for 24 hours until pale purple thin films were formed. By increasing the drying time, the SPE films had shifted to colourless. From this, it could be considered that the changes in SPE film features might alter the structure and its morphology. Moreover, the difference in colour or transparency might be caused by the changes in internal structures developed during the drying of the films [28]. The thickness of the SPE films was measured in a range of 0.104 mm to 0.124 mm. It slightly varied due to the viscosity of the polymer solutions.

The XRD is a technique used to identify the changes of phases in SPE film whether in amorphous or crystalline regions [29]. Figure 2 displays the XRD of SPE films. From the results, it can be seen that the pure corn starch film showed a broad hump at 15° to 28° , which corresponded to the amorphous nature of the polymer [30]. Furthermore, as seen in all patterns, the addition of NaIO_3 into the polymer electrolyte would give no significant change except for the higher salt content. Specifically, with 1 wt. % of NaIO_3 , the pattern of complex films changed to a wider hump as compared to the pure corn starch film with 68% of amorphicity. Then, the incorporation of 3 wt.% of NaIO_3 showed a pattern similar to the complex film that contained 1 wt. % of NaIO_3 but the amorphous phase had increased to 77%. The amorphicity started to decrease with a further addition of NaIO_3 . This might be caused by the interaction between the sodium salt and polymer host, which altered the film's state. It could be seen that the amorphousness of the SPE films started to decrease for samples containing 5 wt.% and 7 wt.% of NaIO_3 with a value of 58% and 40%, respectively. Thereafter, several crystalline peaks appeared at $2\theta = 14.3^\circ$, 21.4° , 24.9° , 27.1° and 32.7° for the SPE sample with 9 wt.% of NaIO_3 . Therefore, from the XRD results, it can be summarised that the SPE films tend to appear in the amorphous phase rather than crystalline phase. Moreover, the presence of NaIO_3 did not significantly influence the formation of peaks in the complex samples.

The morphology and microstructure of SPE films were investigated by using the scanning electron microscopy (SEM). Figure 3 shows the surface images of the SPE films. In general, the surface condition played a vital role in the enhancement of conductivity and was linked with an amorphous content. In addition, according to [31] and [32], the smooth surface of the films would provide a better ionic conductivity, which correlates to the amorphous nature of the sample. It could be observed that the surface of the films became rougher with the addition of salt. The increase in the roughness and amorphous content was responsible for promoting the ion movement, hence, enhancing the conductivity. Therefore, it can be concluded that when salt was added to the polymer electrolyte, the

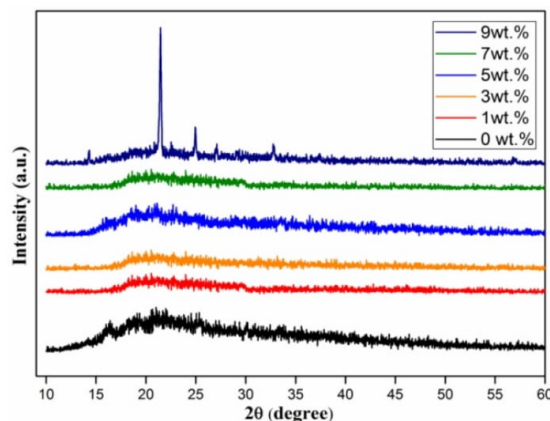


FIGURE 2. The X-ray diffraction pattern of corn starch doped with sodium iodate (NaIO_3).

film surface was slightly modified, which indicated that the salt was taking part in the system.

Figure 4 shows the SEM images of the prepared films. The corn starch image (Figure 4a) revealed the smooth surface and compact structure without any pores or cracks. Upon addition of 1 wt.% NaIO_3 (Figure 4b), the rough images appeared. It was clear from Figure 4b that after the addition of sodium salt, the surface morphology of the SPE film changed. After increasing the salt content to 3 wt.% (Figure 4c), the film images showed a higher number of pores with a larger diameter. It was believed that the appearance of pores could help the ions to penetrate easily through the membrane. Thus, it could lead to a rise in the conductivity. The branched structure with some pores are presented in Figure 4d (5 wt.% NaIO_3). From the figure, the number and estimated pore sizes were smaller than in the case of the previous SPE film sample. Furthermore, for SPE films that contained 7 wt.% of NaIO_3 (Figure 4e) the root image did not show any salt particles present. The SPE film with the highest content of NaIO_3 (9 wt.%) appeared with some impurities and tore on the surface.

The diameter of pores for selected SPE films (Figure 4c - Figure 4d) was analysed using ImageJ software and the results are shown in Figure 5. For the SPE film with 3 wt.% of NaIO_3 , the diameter size is in the range of 5.00 to 26.01 μm . As stated by [33], the characteristics of pores are also to enhance the conductivity values because they could assist the ions to easier penetrate through the polymer matrix. Moreover, the larger pore size will also contribute to the higher ion penetration through the system for ion conduction. Hence, the conductivity of SPE films will increase. This is confirmed by the fact that higher conductivity values were obtained with a higher number and larger size of pores. Then, 5 wt.% of NaIO_3 SPE film has shown a decrease in the amount and diameter of pores between 7.61 to 13.15 μm .

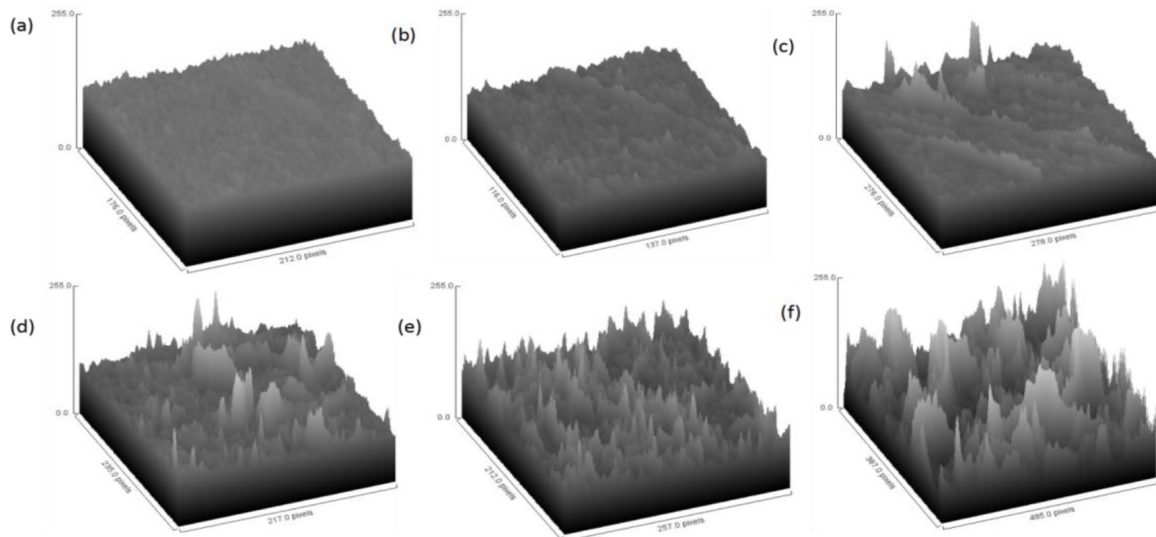


FIGURE 3. The surface conditions of SPE films that contain various ratios of NaIO₃ (a) 0 wt.%, (b) 1 wt.%, (c) 3 wt.%, (d) 5 wt.%, (e) 7 wt.% and (f) 9 wt %.

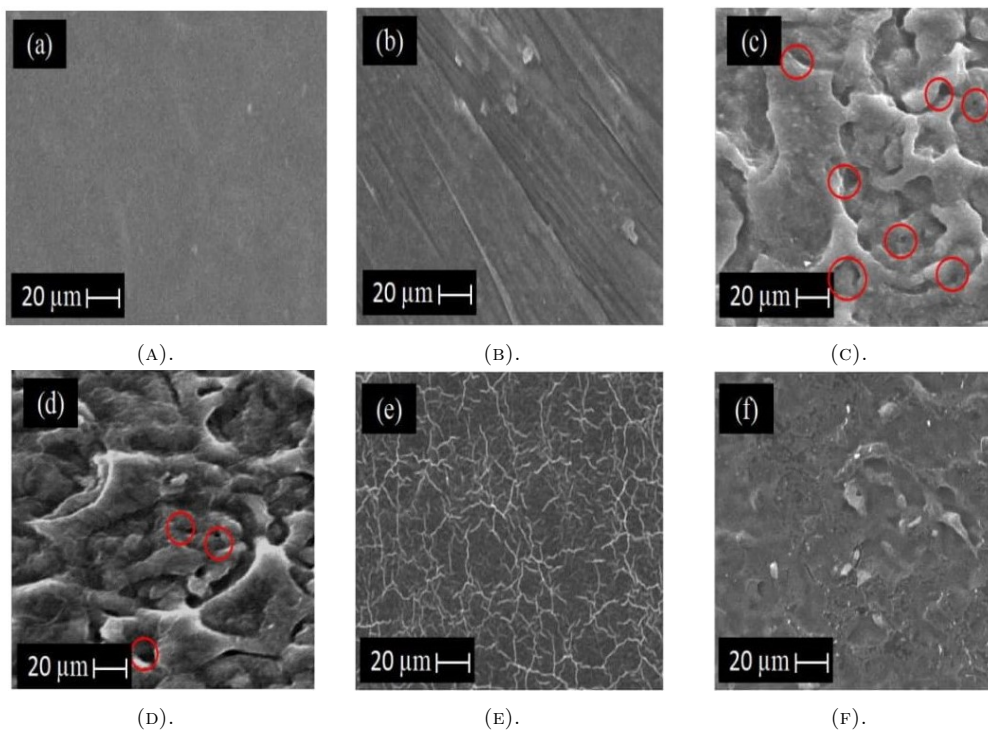


FIGURE 4. SEM micrographs of the surfaces for (a) corn starch film, (b) 1 wt.% of NaIO₃, (c) 3 wt.% of NaIO₃, (d) 5 wt.% of NaIO₃, (e) 7 wt.% of NaIO₃, (f) 9 wt.% of NaIO₃.

Designation	NaIO ₃ concentration (wt. %)	Bulk resistance, R _b (Ω)
A	0	3.00 × 10 ³
B	1	2.00 × 10 ²
C	3	4.70 × 10 ¹
D	5	1.50 × 10 ²
E	7	3.32 × 10 ²
F	9	5.00 × 10 ²

TABLE 1. The bulk resistance and conductivity of SPE films at different salt concentrations.

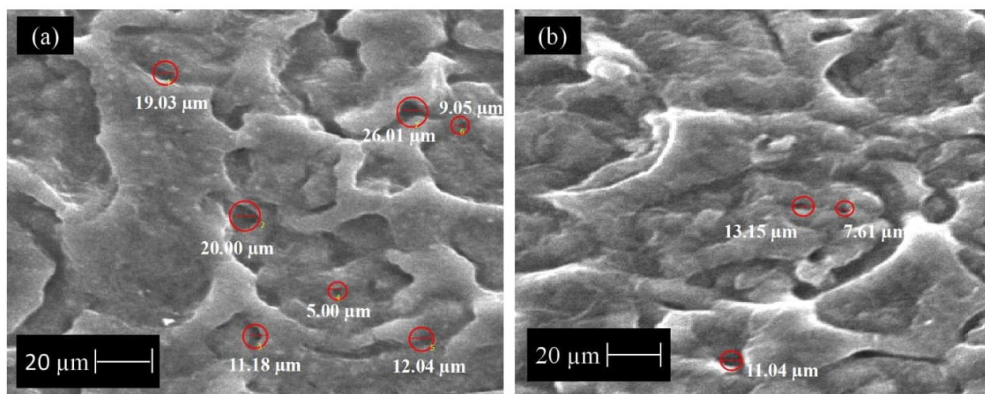


FIGURE 5. The SEM image of a pore in the specific location for (a) 3 wt.% of NaIO_3 and (b) 5 wt.% of NaIO_3 content in SPE films .

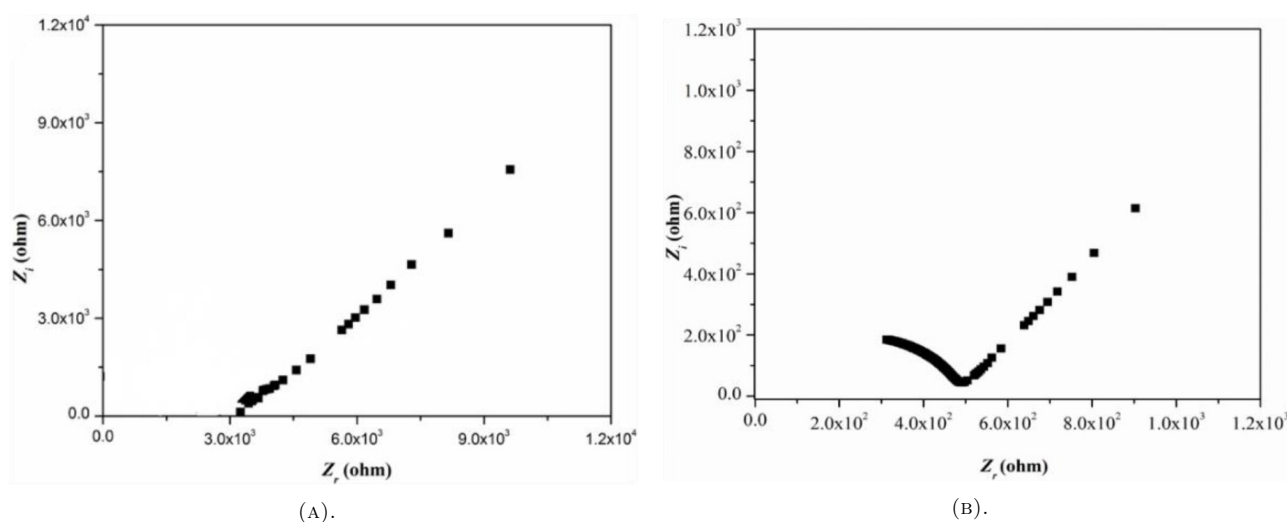


FIGURE 6. Cole-Cole plots for (a) 0 wt. % of NaIO_3 and (b) 9 wt. % of NaIO_3 SPE films.

The Nyquist plot or Cole-Cole plot is a powerful technique used to better understand the electrical properties of the SPE films. Typically, the Cole-Cole plot is comprised of two semicircles. The first semicircle forms due to the contribution of the grain boundary at a low frequency and the second forms due to the grain or bulk properties at a high frequency of the material. In general, the bulk resistance (R_b) value of the sample is determined from the plot in the interception of the higher frequency region on the Z_r axis. Figure 6 shows the Cole-Cole plot of SPE samples containing 0 and 9 wt. % of NaIO_3 while the bulk resistance values are tabulated in Table 1. A reduction of bulk resistance (R_b) also might affect the ionic conductivity values. As the conductivity of SPE films is calculated using Equation 3, as stated before.

The best way to study the properties and mechanisms of any electrochemical reaction is by applying complex impedance spectroscopy [34]. It was observed that the conductivity of pure corn starch film was $1.1 \times 10^{-6} \text{ Scm}^{-1}$. The conductivity started to increase when the sodium salt was introduced into the electrolyte system. The conductivity increased to $1.08 \times$

10^{-4} Scm^{-1} with the presence of 3 wt.% of NaIO_3 . It could be concluded that the rise in conductivity, as the salt content increased, was attributed to the increase in ion concentration in the polymer matrix [35]. Additionally, the increase in the ion density would raise the number of free ions, which could lead to an increase in conductivity.

Further addition of salt into the electrolyte caused a decrease in conductivity. It might be due to the inability of the salt to fill the polymer host and deposit it on the surface after the films were formed [36]. This might decrease the number of mobile ions in the SPE sample, thus decreasing the conductivity. Moreover, [37] also reported that at a higher salt concentration, the distance between dissociated ions may become too small, which leads to the ions reassociating back to neutral ion pairs that do not contribute to the ion conduction. This phenomenon could be observed at a higher NaIO_3 , 5 wt. % - 9 wt. % concentrations in the electrolyte will decrease the conductivity values. Figure 7 indicates the percentage of crystallinity versus conductivity for the SPE films.

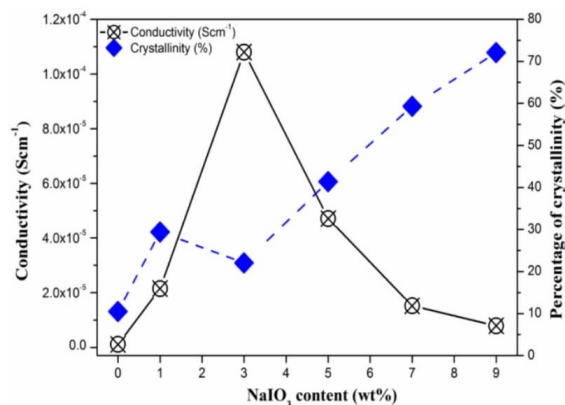


FIGURE 7. The crystallinity (%) against conductivity for each SPE film.

4. CONCLUSION

In conclusion, solid polymer electrolytes based on corn starch-NaIO₃ were successfully prepared using the solution cast method. The XRD analysis proved the coexistence of peaks, which confirmed the complexation of studied materials took place. From the SEM results, it showed that doping sodium iodate with corn starch improved the structural integrity. Moreover, it can be seen that the SPE film, which contained 3 wt. % of NaIO₃ ($1.08 \times 10^{-4} \text{ Scm}^{-1}$) tends to have a branch structure with some pores to enable it to facilitate the movement of free ions through the polymer electrolyte. It was also believed that the prepared SPE film had the potential to be used for energy storage applications such as in batteries as the required minimum conductivity value was achieved.

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