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Mechanical properties and ionic conductivity of biodegradable materials in solid polymer electrolyte

Fatin Farhana AWANG[1], Mohd Faiz HASSAN[1,*], and Khadijah Hilmun KAMARUDIN[1]

[1]*Advanced Nano Materials (ANoMa) Research Group, Faculty of Science and Marine Environment, University Malaysia Terengganu, 21030 Kuala Nerus, Terengganu, Malaysia*

***Corresponding author e-mail:** mfhassan@umt.edu.my

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Abstract

In this paper, the mechanical properties of corn starch-sodium bisulfite ($NaHSO_3$) solid polymer electrolyte (SPE) were investigated. The SPE film based on corn starch was doped with different weight percentages (wt%) of $NaHSO_3$ and prepared using a solution casting method. The SPE was tested by using the Tensile Analyzer to determine the tensile strength and Young's modulus value. The presence of 5 wt% of $NaHSO_3$ content within the corn starch matrix increased the mechanical properties of SPE film from 0.26 MPa to 2.11 MPa of tensile strength while Young's modulus enlarged from 1.6×10^{-2} up to 2.6×10^{-2} MPa. There was found that the tensile strength and Young's modulus values tended to decrease with the addition of $NaHSO_3$ more than 5 wt%. This study highlighted that adding $NaHSO_3$ not only improved the ionic conductivity but also changed the mechanical properties of the film itself and it is believed that these properties had the potential and beneficial not only in advanced electronic applications but also in the packaging industry.

1. Introduction

In recent years, the usage of solid polymer electrolytes (SPE) has grown in demand compared to liquid electrolytes due to the evolutional in electrochemical devices specifically in rechargeable batteries, solar cells, supercapacitors, sensors and semiconductors [1-5]. Commonly, most of the previous works used an inorganic polymer and inorganic solvent as starting materials in SPE film preparation. Nowadays, the usage of natural polymers such as starch, chitosan and cellulose had gained much attention among researchers due to its nature properties (environmental friendly and inexpensive) [6-8]. The selection of relevant initial materials in SPE production at the major stage can directly improve the physical, chemical and electrical properties of SPE film itself and also could cut the cost of production.

Solid polymer electrolytes (SPE) are well-known to have a lot of advantages such as a high capability to eliminate leakage problems, light in weight, easier to shape and mechanically stable [9-16]. The requirement for SPE to be used in certain devices must have high flexibility in its mechanical properties and at the same time the electrical and other properties did not deteriorate [17]. Furthermore, the research regarding the corn starch based SPE for sodium battery separator was still few reported. In addition, most of the works done by researchers that related to this SPE system did not discuss and elaborate in detail on the mechanical section, which is a good characteristic to be looking after the ionic conductivity feature.

In this work, starch-based electrolytes have been prepared by doped with $NaHSO_3$ through a solution casting method. This study highlighted the physical properties of SPE systems through the mechanical and conductivity results reported here. The addition of ionic salts in polymer electrolytes is one of the possible ways to enhance conductivity. The selection of $NaHSO_3$ in this study, instead of it rarely reported, its characteristics like low toxicity, ready availability, lightweight and low atomic mass make it highly promising as an ionic booster, specifically in electrochemical applications [18-25]. Besides, a comparative study reported that sodium based SPE possessed higher conductivity compared to lithium based electrolyte at room temperature [26]. To this point, there is no polymer electrolyte prepared from corn starch doped with $NaHSO_3$ to be applied as a solid electrolyte in any energy storage devices. Besides, the differences of this study compared to other reported work are more detailed concerning about mechanical and ionic conductivity of corn starch-$NaHSO_3$ SPE films and based on resulting data then it may be concluded whether these SPE films have the potential to be applied in sodium battery system as a conduction medium. The as-prepared SPEs will be investigated using a tensile analyzer in aiming to get the information on its mechanical properties by applying some forces on the film.

2. Materials and methods

2.1 Materials

The materials used in the preparation of SPE were corn starch ($C_6H_{10}O_5$, Sigma-Aldrich) as a host and sodium bisulfite ($NaHSO_3$-96% pu., Sigma-Aldrich) as an ionic dopant. The distilled water (100% pu.) and glycerine (96% pu.) were used as the mixed solvent to dissolve the corn starch and sodium salt.

2.2 Sample preparation

The common method used to prepare the solid polymer electrolyte (SPE) by using a solution casting technique. Sodium bisulfite ($NaHSO_3$) with different masses is stirred with two different solvents (glycerine and distilled water) until no left residue. Next, corn starch was added into the mixed solution and stirred on the hotplate magnetic stirrer to form a homogenous solution. The solution was then poured into a petri dish and left it dry naturally at room temperature to form electrolyte films. For further drying, the samples were kept into the desiccator that was filled with silica gel. The amount of sodium bisulfite used was listed in Table 1 and the preparation step is shown in Figure 1.

2.3 Measurement of thickness

The thickness of corn starch-$NaHSO_3$ was measured by using a digital micrometre screw gauge (Kincrome 5610). Figure 2 depicts the thickness of SPE films being measured and before that, the SPE films will be cut into a particular dimension (4 cm × 4 cm). Five readings for each specimen films were taken. The reading was taken at the edges and middle regions of the samples.

2.4 Conductivity properties

The ionic conduction of solid polymer electrolyte (SPE) systems was analyzed using electrochemical impedance spectroscopy (EIS). The prepared samples were cut into a suitable size and sandwiched between two stainless blocking electrodes. The impedance measurements were performed using HIOKI 3532-50 LCR Hi-Tester that interfaced with a computer at room temperature. The data were collected at a frequency range of 50 Hz to 1 MHz. The membrane resistance value was taken from the high frequency intercept on the real impedance axis of the Cole- Cole plot. The conductivity values were calculated using Equation (1).

$$\sigma = \frac{l}{R_b A} \qquad (1)$$

Where, σ is the conductivity (Scm^{-1}), l is the thickness of SPE samples (cm), R_b is the bulk resistance in ohm and A is the sample-electrode contact area (cm^2).

2.5 Mechanical properties

The mechanical strength of the SPE film was examined at room temperature by using a Shimadzu Ez-500NLX tensile tester machine. The film was cut into rectangular with a dimension of 3 cm × 1 cm (length × width). The film membrane was tested at a rate of 1 cm·min^{-1}. Each sample was placed as shown in Figure 3 for the tensile test. At initial, the film was clamped between the sample holders. After pressure was applied, the film had changed and its shape became thinner in the middle and the reading start to be observed, which showed by the computer that connected to the machine.

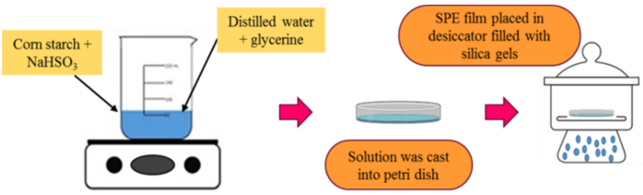

Figure 1. The steps of SPE film preparation.

Figure 2. The thickness of SPE films are measured.

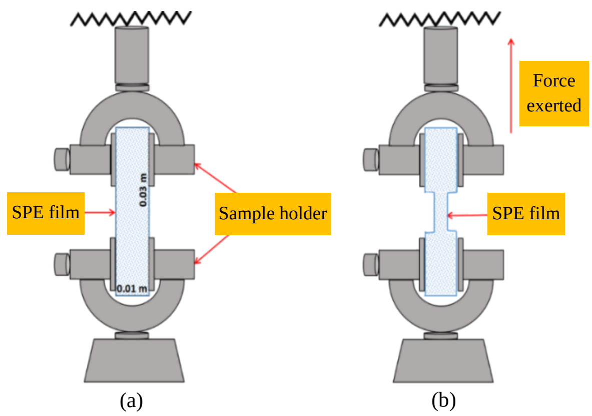

Figure 3. The placement of the thin films SPE for mechanical testing (a) before (b) after.

Table 1. The composition of $NaHSO_3$ used in producing SPE.

Sample	Corn starch (g)	$NaHSO_3$ (wt%)	$NaHSO_3$ (g)	Solvents (mL)	
				Distilled water	Glycerine
Pure	1	0	0	20	0.6
1	1	5	0.053	20	0.6
2	1	10	0.111	20	0.6
3	1	15	0.176	20	0.6
4	1	20	0.250	20	0.6
5	1	25	0.333	20	0.6

In mechanical properties of materials, the tensile strength and Young's Modulus are two important parameters which the ability of materials to resist the specific force and at the same time can maintain other properties at great performance. Tensile strength is a parameter in which to know the limitation of material to withstand stretching before fracture. Whereas, Young's modulus is defined as an elastic modulus that can determine the deformation that happens for materials when a force is applied parallel to an object. In other words, it is used to measure the stiffness of the materials [27-29]. Ductility is the physical property to determine the ability of materials to sustain without breaking even though the shape was changed. The following Equations (2), (3) and (4) are used to calculate the mechanical properties of the studied films.

To get the tensile strength, the force (*N*) which applies to break the film must be divided by the original width (m) and thickness (m) of the film. In mathematical form, the tensile strength formula can be shown by Equation (2) as follows:

$$TS = \frac{load\ at\ break}{(original\ width)(original\ thickness)} \quad (2)$$

Young's modulus is sometimes referred to as the modulus of elasticity and it can be calculated by the tension stress divided by the extensional strain of the film. In other forms, it can be summarized by the following Equation (3).

$$E = \frac{tensile\ stress}{extensional\ strain} = \frac{Fl_o}{A_o \Delta l} \quad (3)$$

Where E is Young's modulus (Pa), F is the force exerted on the object (N), L_o is the initial length of the object (m), A_o is the original cross-sectional area which the area that force applied during the test (m^2) and ΔL is the difference value of length for the object before and after tests (m).

The ductility of material can be represented by the percent elongation of the object. To get it, the difference length in between l_f and l_o divided by l_o time 100%. The mathematical formula for the ductility of materials is summarized in Equation (4).

$$\%\ elongation = \frac{l_f - l_o}{l_o} \times 100\% \quad (4)$$

Where l_f is the length between gage marks after the SPE films break (m) and l_o is the gauge length of the SPE films (m).

3. Results and discussion

3.1 The physical images of selected SPE thin films

The images of selected SPE films with various amounts of ionic salts are illustrated in Figure 4. The images were taken by using a mobile phone camera with 8 MP and 5x of magnifications. It shows that the pure SPE film was transparent. But then, the films had changed into translucent with the addition of $NaHSO_3$ (5 wt%, 15 wt%, and 25 wt%). The changes physical structure of SPE films corresponding with the number of salts added will also have potential to alter the morphology respectively [30]. Besides, Ahmad and his team also reported that the addition of salt may influence the surface morphology of the films to make it become more flexible [31].

3.2 The thickness and conductivity of SPEs

Table 2 shows the thickness reading of SPE. The reading was taken a few times and the average value of measurement was used to calculate the desirable parameters and be plotted in a graph. Figure 5 shows a relationship graph of thickness and conductivity versus the salt content. Briefly, the thickness increases with addition of $NaHSO_3$ salt in between 5 wt% and 25 wt%. The thinnest film in this study obtained from the film containing 5 wt% of $NaHSO_3$ whereas the film with 25 wt% of $NaHSO_3$ gives the thickest film. The measured thickness is attained in the range of 1.01×10^{-2} cm to 2.93×10^{-2} cm. Meanwhile, at 0 wt% of $NaHSO_3$ shows the lowest of conductivity value (1.1×10^{-6} Scm^{-1}) and the highest value is 2.22×10^{-4} Scm^{-1} at 15 wt% of $NaHSO_3$. The thickness is detected to have one trend compared to two trends for conductivity. The thickness trend only follows the first trend and opposes the second trend of conductivity. The two trends of conductivity obtained in two places which are for an increased trend, it starts from 0 wt% to 15 wt% and for a decrease trend can be found from above 15 wt% to 25 wt%. The differential in the thickness of films could be influenced by two main factors, the first, is due to the volume of SPE solution that poured into petri dishes and the second, is because of the duration of the evaporation process [32].

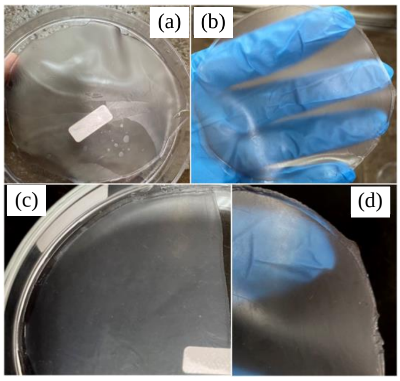

Figure 4. The SPE films of (a) pure corn starch (b) 5 wt% of $NaHSO_3$, (c) 15 wt% of $NaHSO_3$ and 25 wt% of $NaHSO_3$.

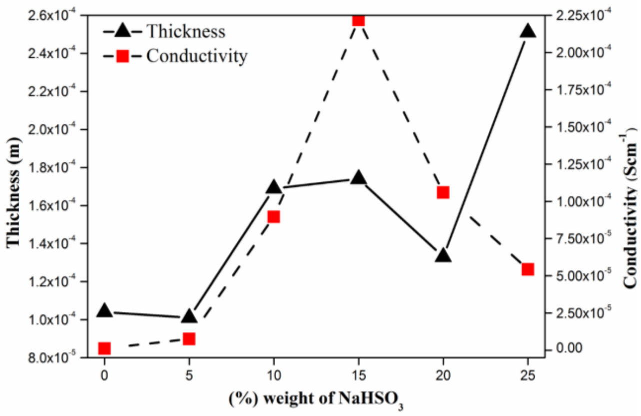

Figure 5. The graph of thickness and conductivity versus the concentration of $NaHSO_3$.

Table 2. The reading thickness of SPE thin films.

$NaHSO_3$ content (wt%)	Reading measurement (cm) ($\pm$ 10^{-5})					Average measurements (cm) ($\pm$ 10^{-5})
	1st	2nd	3rd	4th	5th	
Pure (0)	0.0103	0.0107	0.0102	0.0097	0.0109	0.0104
5	0.0088	0.0122	0.0099	0.0101	0.0097	0.0101
10	0.0180	0.0175	0.0159	0.0173	0.0159	0.0169
15	0.0173	0.0165	0.0173	0.0186	0.0172	0.0174
20	0.0159	0.0156	0.0140	0.0105	0.0105	0.0133
25	0.0266	0.0259	0.0195	0.0261	0.0276	0.0251

3.3 Mechanical properties

Figure 6 presents the tensile strength of corn starch-$NaHSO_3$ SPE against the concentration of $NaHSO_3$. A tensile test is useful in aim to determine the strength characteristics of tested material [33]. The lowest tensile strength is 0.25 MPa at the film without salt and the highest tensile strength of SPE film is recorded at 5 wt% of the $NaHSO_3$ with a value of 2.11 MPa. These values continue to decrease to the second lowest (0.65 MPa) at 25 wt% of salt. With addition 5 wt% of salt, the tensile strength is seen to have an increased trend and further addition salt content its trend tends to decrease. The tensile strength trend follows the trend of conductivity up to addition of 5 wt% salt, and afterward the trend continuously decreases down with addition of 25 wt% salt. It can be understood that the presence of sodium ions into the polymer matrix will lead to improve the tensile strength of the bare film. This happens due to the particles of sodium (Na) ions that can fill the matrix of SPE membranes which led to the denser matrix. So, with some amount of salt it can be seen that the strength of the matrix would be improved at specific parameters. However, when the excess salt (more than 5 wt%) is added to the polymer electrolyte systems, the tensile strength starts to decrease and this decrease trend can be observed further until 25 wt% of $NaHSO_3$. It is believed that the decrease in the tensile strength of the complexes SPE films due to the effect of excess amount of sodium bisulfite which play a major role to modify the core structure of the system to be more crystal and brittle. This happens due to the interruption between salt and intermolecular polymer host to form new weak interactions which can decrease the mixing entropy. The decreased entropy of mixing will make the polymer membrane become less flexible and hard to manage. Thus, decreasing the molecular movements would increase the crystallinity and reduce the conductivity.

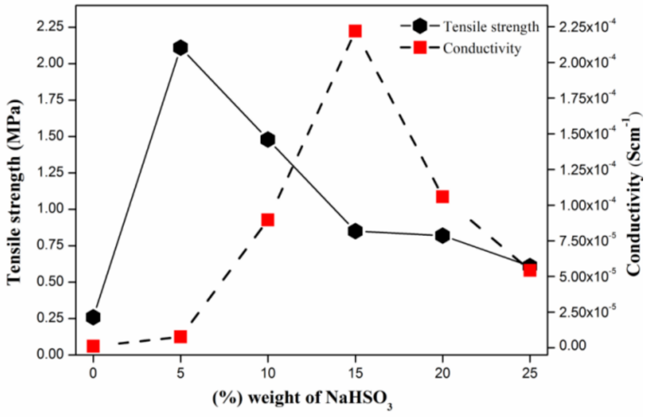

Figure 6. The tensile strength and conductivity of corn starch-$NaHSO_3$ versus the wt.% of $NaHSO_3$.

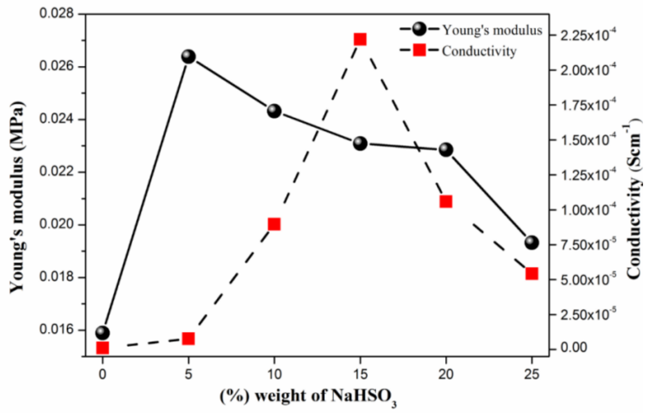

Figure 7. The Young's modulus and conductivity versus salt content graph.

Young's modulus and conductivity versus to different concentrations of $NaHSO_3$ is shown in Figure 7. The pattern of Young's modulus is similar to the pattern of tensile strength with the highest value of Young's modulus of SPE film is 2.6×10^{-2} MPa achieved in adding 5 wt% of $NaHSO_3$. Further addition of sodium bisulfite (up to 25 wt%), the Young's modulus value continuously decreases down near to 0.020 MPa. The increased trend of elasticity in this study can be explained that the addition of sodium salt in the bare film would not only increase the ionic conductivity property but also could improve the properties of amorphicity and enhance the flexibility of the film membrane [34]. However, with excess amount of salt in the system, it could also reduce the specific aspects which are measured, especially on their ionic conductivity and mechanical properties. In this particular study, it can be seen that the film starts to have some good elasticity properties after addition of 5 wt% and further adding salt (up to 15 wt%) their elasticity feature stays at near constant plateau value. During this, the ionic conductivity of the film is at the highest value. With more salt in the system, the elasticity of film begins to give lower value and is similar to the conductivity trend.

The ductility of the material has been determined using the equation (4) by getting the percentage of elongation for every SPE film. The elongation at break for pure corn starch is 4.7%. Then, the percentage is increased with the increase in sodium bisulfite content in the SPE film. This can be observed when the percentage is 26.9% corresponded to the corn starch doped with 5 wt% of $NaHSO_3$. This value was considered to be high compared to the other research.

Table 3. Comparison of the mechanical properties of corn starch-$NaHSO_3$ prepared films in this work with other systems that were recorded.

System	Tensile strength (MPa)	Young's modulus (MPa)	Elongation at break (%)	Conductivity, σ (Scm^{-1})	References
Corn starch- $NaHSO_3$	2.11	0.026	26.9	2.22×10^{-4}	This work
PEO-MnO_2 (CPE)	1.27	-	-	1.95×10^{-5}	[34]
PEO-LiTFSI (SPE)	2.3 times lower than PEO-MnO_2 CPE (1.27)	-	-	1.38×10^{-5}	[34]
Pure PEO	-	1000	-	2×10^{-7}	[35]
PEO- $LiClO_4$	-	321	-	-	[35]
PEO- alumina	-	1415	-	-	[35]
Starch-$LiClO_4$-Glycerol	0.91 ± 0.06	1.15 ± 0.05	82.65 ± 1.3	7×10^{-4}	[36]
Cellulose acetate- $LiClO_4$	19.89 to 43.29	347.63	2.55 to 4.53	1.79×10^{-4}	[37]
Pure PEO	0.99	-	509	1.5×10^{-7}	[38]
	1.02		457	4.6×10^{-5}	
	1.54		278	-	
Cellulose acetate	19.89	1072.46	2.55	-	[39]
Cellulose acetate-$LiClO_4$	43.29	1753.69	4.52	4.90×10^{-3}	[39]
Cellulose acetate-$LiClO_4$-cellulose succinate	28.03	1284.12	2.85	1.35×10^{-5}	[39]

It can be seen that the addition of a small amount $NaHSO_3$ to the SPE not only increases the conductivity but also improves mechanical stability. Moreover, the elongation of films starts to decrease with values 20.2% and 3.6% correlate to the addition of 10 wt% to 15 wt% of $NaHSO_3$. The addition of $NaHSO_3$ (20 wt% and 25 wt%) into SPE would then influence the increase in the percentage elongation of the samples with values of 15.9% and 26.5%.

As for comparison purposes, the values of mechanical properties and conductivity of this research with other studies are tabulated in Table 3. It can be observed that the result shows for corn starch-$NaHSO_3$ films had a high conductivity and comparable mechanical properties (TS = 2.11, E = 0.026 and elongation (%) = 26.9). Then, it can be said that the tensile characteristics for most of the systems would influence the conductivity values. These results are crucial as a reference for future works.

4. Conclusions

In summary, a solid polymer electrolyte has been successfully prepared by adding $NaHSO_3$ salt in corn starch mixture via the solution casting technique. The films appeared in translucent. The presence of 5 wt% of $NaHSO_3$ content within the corn starch matrix increased the mechanical properties of SPE film from 0.26 MPa to 2.11 MPa of tensile strength while Young's modulus enlarged from 1.6×10^{-2} up to 2.6×10^{-2} MPa. There was found that the tensile strength and Young's modulus values tended to decrease with addition of $NaHSO_3$ more than 5 wt%. This study emphasised that the adding $NaHSO_3$ not only enhanced the electrical properties but it also altered the mechanical properties of the film itself and it is understood that these properties had a potential and advantageous not only in advanced electronic applications but also in the packaging industry.

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