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Solid Polymer Electrolytes Based on Starch-Magnesium Sulphate: Study on Morphology and Electrical Conductivity

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Solid polymer electrolytes based on starch as a polymer host and Magnesium Sulphate ($MgSO_4$) as an ionic dopant were successfully prepared by a single-solvent via solution casting technique. As determined by XRD and FTIR analyses, the solid polymer electrolyte films were in amorphous phase and the coexistence of peaks of the materials (starch and $MgSO_4$) confirming that the complex films were successfully obtained. The SEM observations showed the films appeared to be rough and flat shape of surface. The highest ionic conductivity (σ) of 8.52 $\times 10^{-5}$ S cm^{-1} was achieved at room temperature (303K) for the sample containing 35 wt.% $MgSO_4$. The presented results revealed that the as-prepared solid polymer electrolyte has the potential as dual functional compound in electrochemical storage application.

Keywords: Solid Polymer Electrolyte; Starch; Magnesium Sulphate; Ionic Conductivity; Impedance Spectroscopy

I. INTRODUCTION

A solid polymer electrolyte (SPE) with high ionic conductivity at room temperature has been an importance subject due to the interest in the all solid-state electrochemical devices development[1]. Attention in this field began after Wright, (1978) reported that the electrical conductivity exists in polyethylene oxide doped with alkali metal salts [2]-[3]. Commonly, the SPE without ionic salts is identified to have poor performance at room temperature which is one of its drawbacks compared to the present conventional liquid/hybrid electrolytes [4]. Currently, several studies on SPE mixed with selected ionic salts have been executed and the research had tremendous results. For example, in PEO-$LiClO_4$ based-systems, the highest ionic conductivity of 1.56 $\times 10^{-3}$ S cm^{-3} at 80°C was gained with 10 wt.% of poly(cyclotriphosphazene-co-4,4-sulfonyldiphenol) [5], whereas for the PVA systems, the maximum ionic conductivity can be reached up to 1.50 $\times 10^{-3}$ S cm^{-1} [6]. Thus far, the greatest ionic conductivity can also be obtained for the PVC systems which is

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around 4.39 $\times 10^{-4}$ S cm^{-1} resulted from adding 30 wt.% lithiumbis-(trifluoromethanesulfonyl) imide, (LiTFSI) [7]. These successful works had proven that high ionic conductivity can be simply achieved, and the elasticity of SPE can be well-maintained by adjusting the dopant content in polymer system [8]-[13]. Consequently, advanced study on new SPE is crucial to explore their potential application as well as their possibility to be used in energy storage devices.

In SPE preparation, starch is used as a host polymer. Starch is a carbohydrate extracted from agricultural raw resources which is widely present in factually thousands of everyday food and non-food applications. Since it is renewable and biodegradable, it is also a perfect raw material as a substitute for fossil-fuel components in numerous chemical applications such as plastics, detergents and glues. It is possible as one of the importance components in energy storage field exclusively as a dual functioning film. In some research works, starch was used as a host polymer to create a high ionic membrane and have been proven that its bare ionic conductivity may possibly enhanced if incorporated with applicable ionic salts [14]-[17].

$MgSO_4$ is an inorganic salt containing magnesium, sulfur and oxygen. It is often encountered as the heptahydrate sulfate mineral epsomite ($MgSO_4 \bullet 7H_2O$), commonly called Epsom salt. It is widely used in many applications such as in beauty product, sport and agriculture sectors [18]. It is freely soluble in water, very soluble in boiling water and sparingly soluble in aqueous ethanol [19]. Earlier studies had proven that the polymers-based complexed with $MgSO_4$ have an acceptable ionic conductivity and employed as electrolyte in all solid-state battery fabrication, making it attractive for further exploration by incorporating it with another polymer [20]-[22].

In present work, new polymer electrolyte films have been prepared by using starch powder complexed with $MgSO_4$. The structural modifications of SPE were investigated through the techniques of X-ray diffraction and Infra-red spectroscopy. The morphology evolutions have been examined via SEM technique at high and low magnification configurations. The electrical conductivity properties were determined using electrochemical impedance spectroscopy.

II. MATERIALS AND METHODS

A. Synthesis of ionic liquids

Sample Preparation

Starch (derived from corn) and $MgSO_4$ with 99.99% purities were used to yield complex membrane through a solution casting technique. Briefly, the solutions were prepared by liquidating 1g of starch powder into 30 ml of glycerin-distilled water solvent (heated at 60 to 70°C). Next, the amounts of $MgSO_4$ (varied from 0 to 45 (in weight percentage, wt.%)) were added to the solutions as planned in Table 1. The blends were stirred constantly using magnetic stirrers until homogenous solutions were acquired. Subsequently, the solutions were transferred into different plastic Petri dishes and were left to evaporate to form solid state films. Successively, the films were reserved in desiccators (with silica gel) for a certain period to reduce water content. A schematic diagram of overall preparation process is described in Figure 1. To note here, all the experimental works were carried out at room temperature.

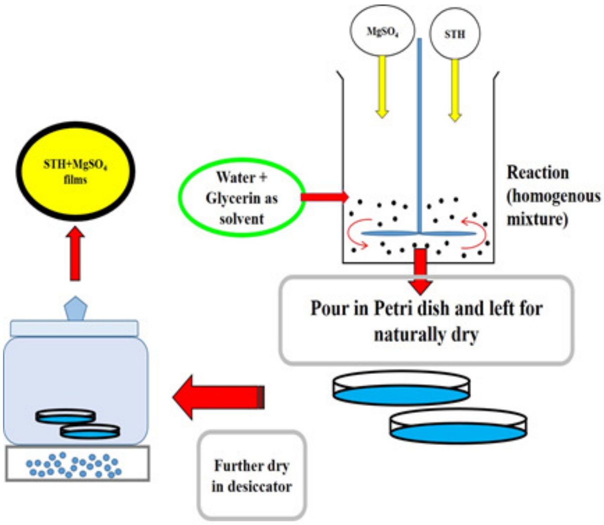

Figure 1. A schematic diagram to prepare starch and starch-$MgSO_4$ complex films.

B. Characterization

X-ray diffraction technique was used in determination of crystallinity of sample. A MiniFlex II diffractometer equipped with an Xcelerator, using CuKα radiation was ran to record XRD patterns in range of $2\Theta = 10^\circ$ to 80°.

The composition and complex formation in the sample was analyzed by a Thermo Nicolet Avatar 380 FT-IR spectrometer. It simultaneously collects spectral data in a wide spectral range from 4000 to 675 cm^{-1} with spectra resolution of 4 cm^{-1}. The spectrometer is equipped with an attenuated total reflection (ATR) accessory with a germanium crystal. The sample was put on the germanium crystal and infrared light was passed through the sample; the graph is sketched.

In order to investigate the effect of $MgSO_4$ on starch surface morphology, scanning electron microscopy was employed to observe the surface images of the sample. It was carried out using the Model JEOL JSM-6360LA device with an acceleration voltage of 20kV. The images were taken for selected magnifications of x300, x5000 and x10000, accordingly. Through this technique, the crystalline or amorphous nature of the sample can be directly observed.

Impedance spectroscopes for room temperature (30°C) were measured using the HIOKI 3532-02 LCR Hi-Tester which is interfaced to a computer. It is used to determine the electrical properties over a wide range of frequencies from 50Hz to 1MHz. The prepared samples were cut into 2 cm diameter size and placed between two stainless steel electrodes on a sample holder which connected via leads to a computer. The bulk resistance, R_b, can be directly obtained from the imaginary impedance (Z_i) versus the real impedance (Z_r) plots from the impedance system. A micrometer-screw gauge was used to measure the sample thickness which will be em-

Table 1. The compositions of starch and $MgSO_4$ powders.

Sample	Solvent (ml)	Starch (g)	$MgSO_4$ (wt.%)	$MgSO_4$ (g)
Pure	30	1	0	0
A	30	1	5	0.053
B	30	1	10	0.111
C	30	1	15	0.177
D	30	1	20	0.250
E	30	1	25	0.333
F	30	1	30	0.429
G	30	1	35	0.548
H	30	1	40	0.667
I	30	1	45	0.834

ployed to calculate conductivity of sample using the Equation (1):

$\sigma = \frac{t}{R_b A}$.......(1)

where t = thickness of the thin film (in cm) and A = area of the contact and R_b= bulk resistance.

III. RESULTS AND DISCUSSIONS

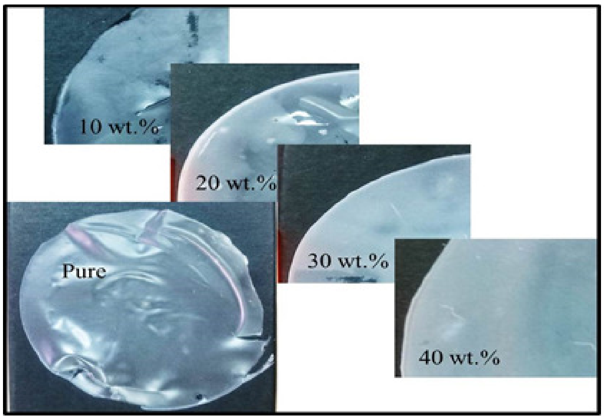

Figure 2. Starch and hybrid starch-$MgSO_4$ films.

Figure 2 shows the images of selected starch-$MgSO_4$ complex films with different amount of ionic salts. With addition of ionic salt, the film had switched from transparent to opaque in response to the amount of salt, potentially change its morphology and structure. According to literature, this phenomenon was due to the presence of crystals which have a size equal to or larger than the wavelength of light. In this case, the $MgSO_4$ salt plays a role to generate a small number of crystal growth and each of them develops to a larger size compared to the wavelength of visible light resulted scatter visible light and inhibit straight propagation thereof. Hence, formed films of transparent become opaque [23]. To be noted, the film based on this complexation only can be obtained with up to 45 wt.% of ionic salt, in excess of that the film was hard to be produced.

The XRD pattern was recorded from 2Θ =

10° to 80° on starch and starch-$MgSO_4$ complex films is depicted in Figure 3. The starch diffraction pattern can be identified with two broad peaks in different location of 2Θ , first is in between 15° to 27°, and the other is in between 27° to 40°. These peaks correspond to the amorphous nature of polymers. The present study on starch diffraction pattern is in good agreement with other studies on similar material as mentioned in literature [24]-[28]. The rest of XRD patterns are referring to starch-$MgSO_4$ complex films. As can be seen from naked eye observations on all patterns, with addition of $MgSO_4$, no significant change can be detected and the films have a certain similarity and presence in amorphous phase. However, a few peaks located at 16.8° and 19.6° were clearly visible in most of starch-$MgSO_4$ diffraction patterns, correspond to $MgSO_4$ peaks. The overall XRD results explained that the complex films incline to presence in amorphous and the coexistence of XRD peaks for the studied materials ratify their complexation in solid polymer electrolyte film [29]-[32].

The FTIR spectra for starch and starch-$MgSO_4$ complex films are displayed in Figure 4. The major bands displayed at 3285, 1657, 1378 and 1024 cm^{-1} were attributed to the starch. The absorption bands at 3285 cm^{-1} corresponded to the O-H stretching vibration, whereas the typical absorption band at 1657 cm^{-1} apparently initiated from strongly bound water existent in the starch molecules [33]-[34].

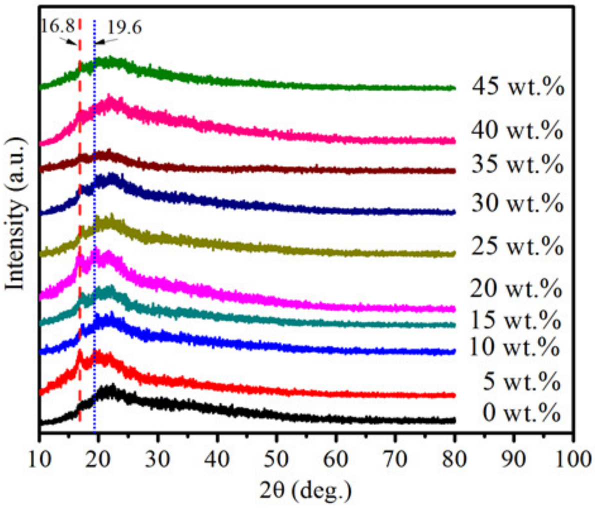

Figure 3. XRD pattern for starch and starch-$MgSO_4$ complex films.

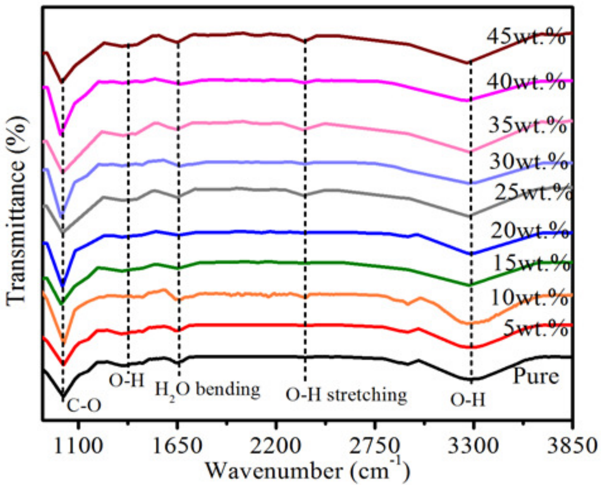

Figure 4. FTIR spectra for starch and starch-$MgSO_4$ complex films.

The peaks at 1378 and 1024 cm^{-1} corresponded to O-H bending and C-O stretching, respectively. These bands were similar to those reported for starch [35],[33]. From the observation on the complex spectra, there were two modified peaks can be detected, first at 2355 cm^{-1} which is with addition of 25 wt.% of $MgSO_4$, the peak started to emerge until 45 wt.% of $MgSO_4$. According to Alagarsamy *et al.* (2018), this peak corresponded to $MgSO_4$ [36]. The other peak is at 2934 cm^{-1} which was corresponds to starch peak and it was disappeared with response to the amount of $MgSO_4$. Coates stated that if an IR spectrum has the functional group of two different materials or any change occurred in spectrum (in this case, starch and $MgSO_4$, it can confirm that the chain-reaction (or addition) polymerization process is successfully achieved due to the existence or modification of each chain in the spectra [37]. By associating the spectrum of starch with those of starch-$MgSO_4$ presented all of the typical bands of starch and $MgSO_4$ and did not illustrate any new bands, which confirmed that no chemical reactions occurred between starch and $MgSO_4$. Table 2 is a summary of the corresponding bands for the studied films. It can be concluded, that the significant modifications in the chemical structure was due to the doping of $MgSO_4$ substantiated that $MgSO_4$ has been inserted into starch in the complexation process.

Figure 5 shows the scanning electron micrograph images of pure starch and starch-$MgSO_4$ complex films. A starch film had a smooth surface morphology (Figure 5a). No different texture was seen with addition of 5 wt.% of $MgSO_4$ (Figure 5b). With addition of 10 to 25 wt.% of $MgSO_4$ (Figure 5c-5f) the film texture was changed with the presence of flat and circle shapes surrounding the area of the film surface and the size was decreased with additional salts. The agglomeration on the top of film can be seen with addition of 30 wt.% of MgSO4 and interestingly, the pores also can be observed at certain areas (Figure 5g). In Figure 5h (35 wt.% of $MgSO_4$), the circle shape turn to be flat, jointly each other′s and the pores structure appear increasing in quantity more than previous sample. The estimated diameter size of pore was in the range of 20 to 70 nm, and was understood that they have special characteristics which one of them could contribute to enhancing the electrical properties of the studied materials [41]-[42]. With addition of 40-45 wt.% of $MgSO_4$, the obvious individual circle shape structure had been produced. It is believed that, this structure type could provide the weak bonding and results the cracked film (Figure 5i and 5j).

The value of bulk resistances, R_b and the ionic conductivity, σ of the samples are tabulated in Table 2, while Figure 6 presents a trend of ionic conductivity variation as a function of salt content at room temperature. The measured thicknesses of the samples were in between 7.4 x 10^{-3} to 1.6 x 10^{-2} cm and used together with the bulk resistance to calculate the ionic conductivity of sample using the Equation (1). The bulk resistance of the pure starch film was 9.82 x $10^2\Omega$ with an ionic conductivity of approximately 1.35 x 10^{-6} S cm^{-1}. With addition of 5 wt.% of $MgSO_4$, the bulk resistance increased to 1.30 x 10^3 Ω and the ionic conductivity in-

Table 2. FTIR absorbance, functional groups and references of starchNaOH complex films

V(cm^{-1}) related crystal system	Functional Group	Reference
1024	(C-O stretching)	He *et al.*,2016 [38]
1378	(O-H bending vibration)	Tongdeesoontorn *et al.*, 2011 [39]
1657	Hydroxyl (H_2O bending)	Mukurumbira *et al.*, 2017 [33]
2355	Attributed to $MgSO_4$	Alagarsamy *et al.* 2018 [36]
2934	Methyl ($-CH_2$ stretching vibration)	Mukurumbira *et al.*, 2017 [33]
3285	Hydroxyl (O-H stretching)	Rachtanapun *et al.*, 2012 [40]

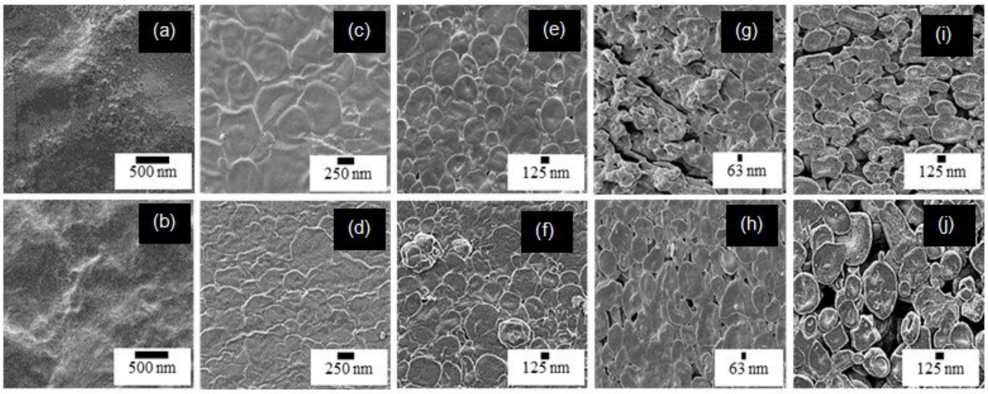

Figure 5. SEM micrographs for (a,) starch film, (b) 5 wt.% $MgSO_4$ (sample A), (c) 10 wt.% $MgSO_4$ (sample B), (d) 15 wt.% $MgSO_4$(sample C), (e) 20 wt.% MgSO4 (sample D), (f) 25 wt.% $MgSO_4$ (sample E), (g) 30 wt.% $MgSO_4$ (sample F), (h) 35 wt.% $MgSO_4$ (sample G), (i) 40 wt.% $MgSO_4$ (sample H) and (j) 45 wt.% MgSO4 (sample I).

creased to 2.10 x 10^{-6} S cm^{-1}. The bulk resistance (7.79 x 10^{-1} Ω) decreased after addition of 10 wt.% $MgSO_4$ and given the ionic conductivity of 2.22 x 10^{-6} S cm^{-1}. With addition of

Table 3. The bulk resistance and ionic conductivity of starch and starchMgSO$_4$ complex films at room temperature.

Sample	Bulk Resistance, R$_b$ (Ω)	Conductivity, σ S cm^{-1}
Pure	9.82 $\times 10^2$	1.35 $\times 10^{-6}$
A	1.30 $\times 10^3$	2.10 $\times 10^{-6}$
B	7.79 x 10^2	2.22 x 10^{-6}
C	1.20 x 10^3	2.59 x 10^{-6}
D	1.26 x 10^3	4.70 x 10^{-6}
E	1.35 x 10^2	2.85 x 10^{-5}
F	4.52 x 10^3	5.55x 10^{-5}
G	3.25 x 10^1	8.52x 10^{-5}
H	9.20x 10^1	5.50x 10^{-5}
I	9.68x 10^1	4.60x 10^{-5}

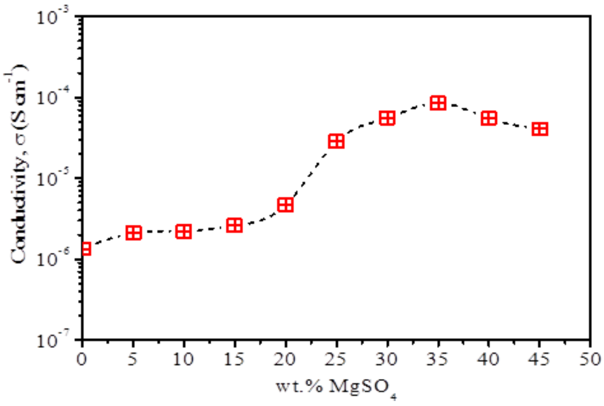

Figure 6. The conductivity variation as a function of salt content at room temperature.

15 wt.% of MgSO$_4$, the bulk resistance increased to 1.20 x 10^3 Ω and the ionic conductivity was 2.59 x 10^{-6} S cm^{-1}. The measured resistance increased to 1.26 x 10^3 Ω with addition of 20 wt.% of MgSO$_4$ and the ionic conductivity was 4.70 x 10^{-6} S cm^{-1}. The increased trend of ionic conductivity continued in 25 to 35 wt.% of MgSO$_4$ with the highest value was 8.52 x 10^{-5}S cm^{-1} which was obtained in 35 wt.% of MgSO$_4$ complex film. With addition of MgSO$_4$ in polymer, experimentally preventing the formation of complex films and further investigation on the electrical conductivity cannot be conducted thru existing equipment.

To summarize, there are two major factors which contribute to the increase trend of ionic conductivity, first is the amorphous structure of complex samples which acts as a supportive medium for fast ionic movement, as reported by a number of researchers, the ionic conductivity in amorphous complex structures is higher than that in crystalline polymer salt complexes due to its relatively low interfacial resistance in between the solid electrolyte and the electrode [43]-[46]. The second factor is related to the inorganic salt which supplies ionic conductors to the closed system until a saturated condition is achieved. This is due to the increase in conductivity with salt content is attributed to the increase in the number of free mobile ions. For the decrease in ionic conductivity trend, it might be due to ion association which decreases the number of free ions available for conduction, or the decrease in mobility of the ions [9]. The drop in conductivity can also be due to the existence of wrecked film and the increase in crystallinity of film which are supported well by XRD and SEM analyses as shown in Figure 3 and Figure 5, respectively. These discoveries are also in good agreement to the investigation conducted by Mellander and

Albinsson, 1996 and Reddy *et al.*, 2007 [47],[45].

IV. SUMMARY

In this work, the ion-conducting solid polymer electrolytes based on starch-$MgSO_4$ have been successfully prepared via a solution casting method. The effects of $MgSO_4$ on the starch structure were investigated on the basis of X-ray diffraction and scanning electron microscope. To our understanding, this innovation of the starch-$MgSO_4$ complex films is the first has been documented, specifically on its structure, morphology and ionic conductivity.

In summary, it can be concluded that the complex films were presence in amorphous state. The XRD and FTIR examinations further proven that there are coexistent peaks attributed to the materials studied on, confirming the success of the complexation development occurring in this film. Agreeing to the morphology observations, the increment of $MgSO_4$ can significantly modify the surface texture of the film, as the highest conducting film containing 35 wt.% of $MgSO_4$ ($8.52 \text{ x } 10^{-5}$ S cm^{-1}) are supported by the amorphous phase and highly number of free mobile ions.

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