

Conductivity Studies and Characterizations of PVA-Orthophosphoric Electrolytes

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Abstract

Solid polymer electrolyte (SPEs) has drawn great attention nowadays, particularly in the field of science and applied science, specifically, those used as separators in industries and researchers for the assemblage of some “classified batteries” and super capacitors. In this report, we present the grandeur of this SPE (pure type), particularly at room temperature, such that, it can be applied as an electrolyte as well as a separator in supercapacitor fabrication. The SPEs were produced from the composition of polyvinyl alcohol (PVA) and Phosphoric acid (H_3PO_4), such that, the PVA samples were kept constant while the phosphoric acid was varied at 0, 10, 20, 30, 40, 50, 60, and 70 wt. %. After the experimentation, the pure polymers show excellent results in term conductivity, in that, it has recorded a conductivity as high as $2.56 \times 10^{-3} \text{ Scm}^{-1}$ at the compositions of 70 wt. %. Aside that, we also observed that, the bulk moduli (R_b) value decreases with the increase in concentration, with pure solid polymer electrolyte (PSPE) recording 2.5Ω at the highest composition of 70 wt. %.

Keywords: Pure solid polymer electrolyte (PSPE), polyvinyl alcohol (PVA), Phosphoric acid (H_3PO_4), Electrochemical Impedance Spectroscopy (EIS).

1. Introduction

The year 2013 marked the complete three decades (1973) of the invention of polymer electrolyte by Fenton and co-workers (Gu et al., 2000; Kumar & Sundari, 2010; Zhang et al., 2006; Yang, 2004; Ragavendran et al., 2004; Tripath et al., 2012; Ulaganathan et al., 2012; Kuo et al., 2013; Yang et al., 2013; Noor et al., 2010) and Armand (Ragavendran et al., 2004; Zhang et al., 2006). Ever since then, research on polymer electrolyte has become very elaborate.

Research has shown that, there are three classifications of polymer electrolyte systems, namely (i) Polyelectrolytes (PEs): - which have an independent ion-generating groups chemically bond to the macromolecular chain and presence of counter-ion in order to maintain the electroneutrality of the salt. In dry conditions, they have low conductivity of about $10^{-10} - 10^{-15} \text{ Scm}^{-1}$ but in the presence of dielectric solvent such as water, there conductivities do improve (Ulaganathan et al., 2012). (ii) Solvent Swollen Polymer Electrolytes (SSPE): - In SSPE, solvents, both aqueous and non-aqueous swells the host polymer. Thus, the dopant ionic solutes such as H_3PO_4 is hosted in the swollen lattice thereby permitting the movement of ions in the solvent swollen region of the polymer host. Their conductivity depends on the compactness of the solvent in the region that is being puffed up (Ulaganathan et al., 2012). And lastly (iii) the Solvent Free Polymer Electrolytes (SFPE): These are polymer salt complex, which is formed by complexes between salts of alkali metals and polymer containing solvating hetroatoms, example; O, S and N (Ulaganathan et al., 2012). The complexation between poly ethylene oxide (PEO) and alkali metal salts is a good example of SFPE. SFPE are sub-classed into three also. They are; (a) Solid Polymer Electrolytes (SPEs), (b) Gel Polymer Electrolytes (GPEs), and (c) Composite polymer electrolytes (CPEs) (Kuo et al., 2013; Tripath et al., 2012).

GPEs can be only considered as plasticizers incorporated polymer-salt complex (Ulaganathan et al., 2012). They are therefore consisted of a liquid electrolytes immobilized in a polymer matrix (Hashmi, (a), 2013) and exhibits high ionic conductivity (10^{-3} to 10^{-4} Scm^{-1} at ambient temperature) and can be obtained by either loading of a liquid electrolyte in a microporous matrix or by increasing the viscosity of a liquid electrolyte through the addition of a soluble polymer until gel consistency is achieved or by mixing a liquid electrolyte with monomers of relatively low molecular weight, then curing the mixture by other means, such as UV polymerization, thermal

polymerization and electron beam radiation polymerization. They are criticized for having weak mechanical strength and poor interracial properties, (Qiu et al., 2004; Hashmi, (b), 2013). In CPEs, they are normally prepared by addition of high surface inorganic fillers like SiO_2 , MgO , TiO_2 and alike so as to improve the mechanical strength and stiffness of the complex systems (Ulaganathan et al., 2012).

SPE is a thin film that has ionic conductivity, especially, when an alkali salt dissolves in polymer matrix. SPE have drawn great attention in the development of science and technology of lithium secondary batteries. SPEs have several advantages over liquid, gel and even composite electrolytes in that, it has process ability, flexibility, light weight, elasticity and transparency (Zhang et al., 2006), desirable shape moldability, being free from leakage, mechanical strength, better stability especially in high temperature, and high specific energy and power (Lim et al., 2012). Furthermore, use of solid polymer electrolyte can negate the need for a separator and be amendable to low cost manufacturing technologies (Qiu et al., 2004). Some of the applications of SPE are in Batteries, fuel cell, supercapacitors and other electrochemical devices. (Sawada et al., 2000) and other electrochemical devices (Sotta et al., 2010).

Although number of works have been carried out on the characterizations of SSPE like those of Prajapati et al., (2010) and Ahmad Khair & Arof, (2010), this piece tries to offer a tone by step approach on the conductivity studies of this transparent polymer in order to ascertain the suitability composition for the usage in the supercapacitor fabrication which is the main target of our future work. Thus, careful measures have been put in place to ensure that every detail characteristic of the active materials, especially of the PVA – its molecular weight and its percentage hydrolysis – were taken into considerations.

2. Experimental

2.1. Preparation of the Composite PVA Electrolytes

The pure solid polymer electrolyte (PSPE) which can also be served as separators in supercapacitors fabrications have their laboratory preparations discussed elsewhere by us, in Hashim et al., (2012), Hashim et al., (2014) and by Jiang et al., (2013). But for the purpose of elaboration; the active materials comprise of H_3PO_4 and PVA. The H_3PO_4 functions as an ionic liquid, while PVA acts as a polymeric matrix, allowing ionic transport while simultaneously functioning as a separator between the electrodes of the supercapacitor. H_3PO_4 (>85 wt. % in water, molar mass of 98.00 g/mol, product, number of 1502-80) was obtained in aqueous form, from R & M marketing, Essex, UK brand, while the PVA (molecular weight; 89,000-98,000, 99+ % hydrolyzed) was obtained from Sigma Aldrich. Both H_3PO_4 and PVA were used as-received without further treatment or purification. An aqueous solution of PVA is prepared by combining PVA with distilled water in the ratio of 1:10 by volume. This solution is mechanically agitated by magnetic stirring at 60 °C for five hours to thoroughly dissolve the PVA in the distilled water. H_3PO_4 was then mixed with the PVA aqueous solution in the ratio of 100:0, 90:10, 80:20, 70:30, 60:40, 50:50, 40:60 and 30:70 wt. % for the PSPE to be obtained (this has been summarized in Table 1).

In order to vary the percentage of the H_3PO_4 used, the following formula was used;

$$\frac{n}{n+2} \times 100\% = x\% \quad (1)$$

Where x is the percentage of acid require (0, 10, 20, 30, 40, 50, 60, and 70 % wt), and n is the value of H_3PO_4 in grams.

The mixing is done in a drop-wise manner with magnetic stirring at 60 °C for about one hour or thereabouts until it completely turns to homogeneous solution (transparent in this case). The mixture is then allowed to cool down to an ambient temperature. The resulting homogeneous solution of PVA/ H_3PO_4 is cast over a plastic Petri dish. Prior to this, the Petri dish was scraped off the dust using a tissue soaked in acetone. The PVA/ H_3PO_4 solution solidifies onto the Petri dish and upon curing for about 3 – 4 weeks at room temperature. This is due to its high molecular weight and consequently more hydrolyzed. The solid layer was easily peeled off from the Petri dish after it dries as a freestanding layer. The thickness of the solid layers formed in this procedure can be controlled depending on the composition that have been enumerated above and as shown in Figure 1. The resulting polymer film was then put in the plastic bags for safe keeping, and to avoid contamination from surrounding, for further property analysis.

3. Result and Discussion

3.1. Conductivity and Chemical Composition Analysis

Conductivity measurements were carried out on both the solid and hybrid polymer electrolyte films which are also composed of all the different percentage ratio of PVA- H_3PO_4 an impedance method which also was highlighted by Ahmad Khair and Arof (2010). The solid polymer films were sandwiched between a stainless steel, ion-blocking

liked measuring device (otherwise known as a probe), each of surface area 2 cm^2 in radius, in a spring-loaded glass holder.

The impedance measurements were carried out by using Electrochemical Impedance Spectroscopy (EIS) testing machine named HIOKI 3532-50 LCR Hi-Tester which was connected to the computer. The frequency range of the device was 50 Hz - 1 MHz, and simultaneously calculating both real and imaginary impedance.

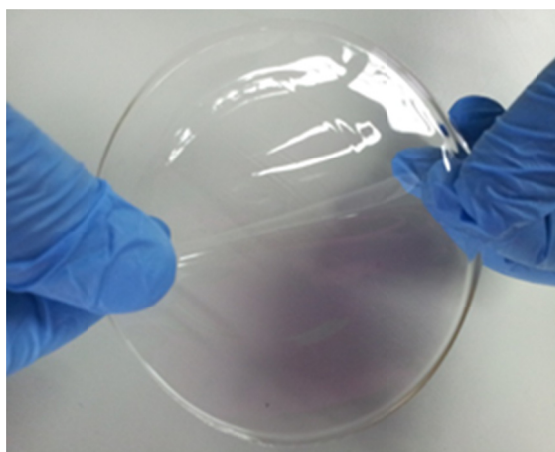


Figure 1. Showing a prepared and flexible PSPE

Table 1. Table Showing the Overall Formation of the PSPE Preparation

Coding	Measured Quantity PVA in Distilled Water (g)	Measured Quantity of H_3PO_4 (g)	Ratio of PVA to H_3PO_4
P0	2	0	100:0
P10	2	0.22	90:10
P20	2	0.50	80:20
P30	2	0.86	70:30
P40	2	1.33	60:40
P50	2	2.00	50:50
P60	2	3.00	40:60
P70	2	4.67	30:70

The sample exhibits Debye characteristics, and then the plots of the negative imaginary impedance versus the real impedance on the graph should be able to display a semicircle-like shape (Ahmad Khair & Arof 2010). From the Eq. 2, we calculated the electrical conductivity of the samples;

$$\sigma = \frac{l}{R_b A} \quad (2)$$

Where σ is the conductivity itself, l and A are respectively the thickness and area of the samples. The thickness of the sample was measured twice at different positions of the polymer films and an average was taken using digital micrometer.

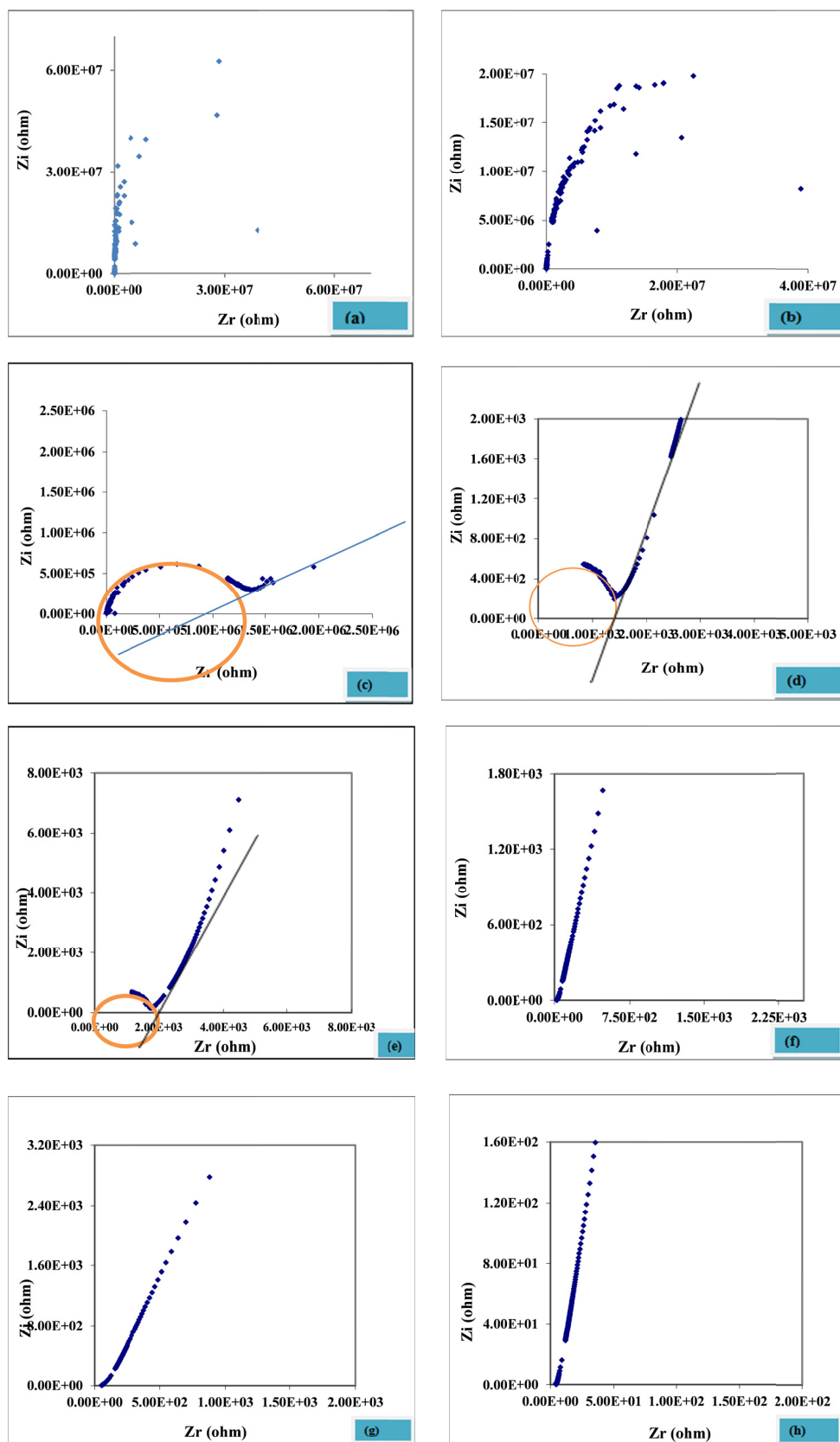


Figure 2. Impedance plots of PSPE containing 0 wt.% (a); 10 wt.% (b); 20 wt.% (c); 30 wt.% (d); 40 wt.% (e); 50 wt.% (f); 60 wt.% (g); 70 wt.% (h) ratio of the H_3PO_4 at room temperatures

Figure 2 (a-h) shows the impedance plots of a sample of the PSPE containing 0, 10, 20, 30, 40, 50, 60, and 70 wt. % ratio of the H_3PO_4 at room temperatures respectively. For the sample with 0 and 10 wt.% of the H_3PO_4 , it can be observed that the point of the graph was dispersed, which clearly indicates the meager or absent of acid in this sample.

The semicircles found on the Figure of the samples containing 20, 30 and 40 wt.% could however be connected with the impact of the introduction of the percentage acid and consequently the immobile polymer chain begins to emerge.

As the acid concentration continues to increase, so the conductivity, thereby making the semicircle disappear, which is an indication that, ions that move around the polymer matrix made resistive component of the polymer electrolytes to exist. (Ahmad Khair & Arof, 2010; Ulaganathan et al., 2012).

The calculated R_b for all the eight samples above were obtained from the frequency intercepts on each and every plotted graph. We also observed that, the R_b value decreases with the increase in concentration. The calculated values of the R_b are 1.15×10^8 , 8.00×10^4 , 2.40×10^4 , 1.70×10^3 , 2.10×10^3 , 1.93×10^1 , 1.26×10^1 and 2.58Ω for 0, 10, 20, 30, 40, 50, 60, and 70 wt.% ratio of the H_3PO_4 respectively. The highest conductivity of PSPE sample was observed at $2.56 \times 10^{-3} \text{ Scm}^{-1}$ when the H_3PO_4 was increased to 70 wt.% while the lowest conductivity of the sample was noticed at $2.25 \times 10^{-11} \text{ Scm}^{-1}$ where the H_3PO_4 percentage was nil. Full details about the average values of the conductivity of PSPE is summarized in Table 2. On a general note, it can be said that, the sample of PSPE performed wonderfully well in terms of both bulk resistance calculation and the conductivity as a whole.

As mentioned earlier, that conductivity is dependent on the concentration of the H_3PO_4 . Table 2 provided us with the information that, there is a relationship between the acid concentration and the polymer matrix. In addition to that, it also be deduce from the table that, the thickness on each sample, also had some impact on the conductivity of the samples. Hence, because of the thinner nature of the films in PSPE, the conductivity seems to be very promising.

Table 2. Parameters that Determine the Conductivity in PSPE

Coding	PVA: H_3PO_4	l (mm \pm 0.005)	R_b (Ω)	σ (Scm^{-1})
P0	100:0	0.00910	1.15×10^8	2.25×10^{-11}
P10	90:10	0.00630	8.00×10^4	7.20×10^{-8}
P20	80:20	0.01100	2.40×10^4	1.46×10^{-7}
P30	70:30	0.05400	1.70×10^3	1.01×10^{-5}
P40	60:40	0.00585	2.10×10^3	8.87×10^{-7}
P50	50:50	0.00745	1.93×10^1	1.23×10^{-4}
P60	40:60	0.02615	1.26×10^1	6.61×10^{-4}
P70	30:70	0.02070	2.58×10^0	2.56×10^{-3}

3.2 Crystal Structure Analysis

The crystal structure analysis of the solid polymer electrolyte films was examined by a Philip X'Pert X-ray diffractometer (XRD) with Cu K_α radiation of wavelength $\lambda=1.54056 \text{ \AA}$ for 2θ angles between 10° and 80° . The XRD analysis was carried out on the PSPE films. After the analysis, the following results were obtained. The analysis of the structure of the PSPE for 0 wt.% (a); 10 wt.% (b); 20 wt.% (c); 30 wt.% (d); 40 wt.% (e); 50 wt.% (f); 60 wt.% (g); 70 wt.% (h) ratio of the H_3PO_4 was shown in Figure 3.

It can be observed from the semi-crystalline peak of the PVA ($\theta=20^\circ$) which also affirmed the argument put across by (Hashmi, (c), 2013) about the semi-crystal behavior of the PVA. The addition of H_3PO_4 causes the decrease in the degree of the crystallinity and consequently the increase in the amorphicity of the material and its conductivity.

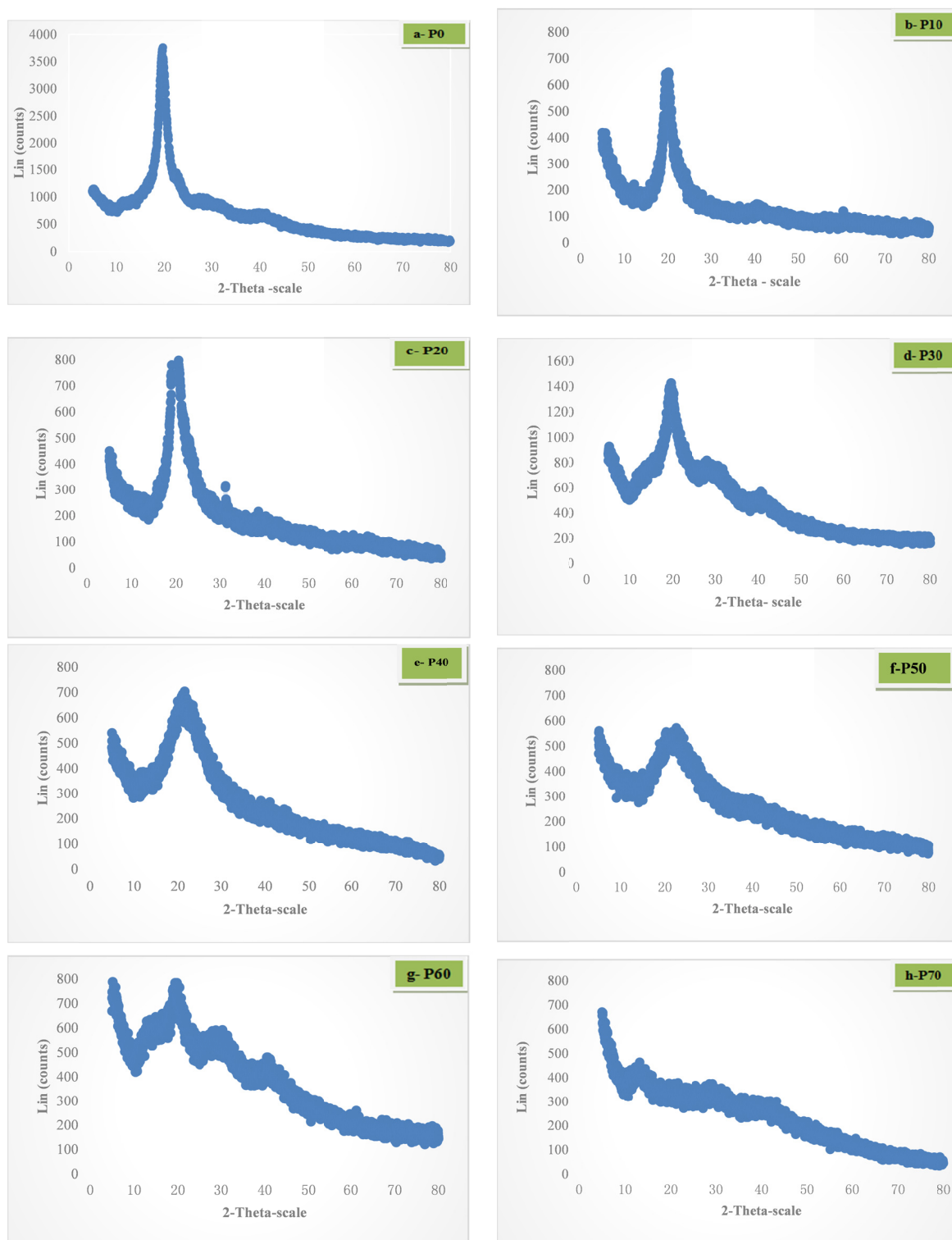


Figure 3. XRD Analysis of PSPE containing 0 wt.% (a); 10 wt.% (b); 20 wt.% (c); 30 wt.% (d); 40 wt.% (e); 50 wt.% (f); 60 wt.% (g); 70 wt.% (h) ratio of the H_3PO_4 and (i) A combined graph containing all the percentages.

3.4 Differential Scanning Calorimetry Analysis

The DSC thermal analysis was carried out using the DCS machine (Mettler Toledo DSC A851 with Star software) under N_2 atmosphere with 10 °C/min heating rate from 40 to 300 °C in order to observe the crystalline and semi-crystalline behavior of the whole samples, therefore, various parameters related to thermal properties such as

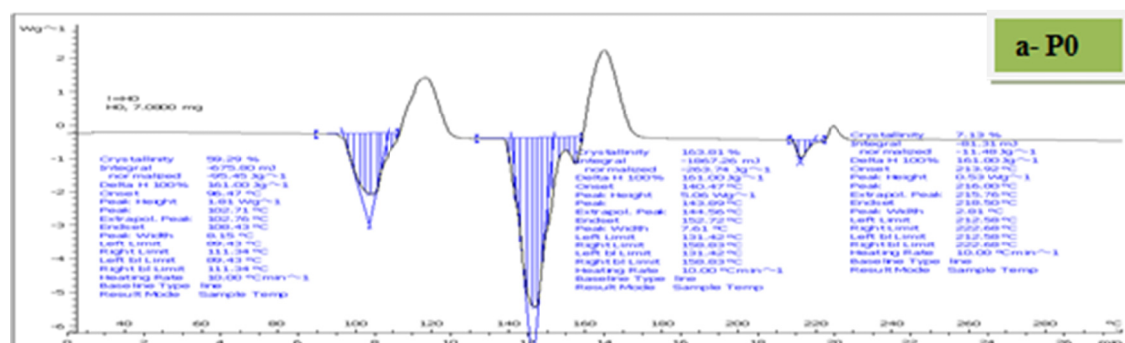
glass transition temperature (T_g), thermal transition (T_m) of crystalline phase of the polymer electrolytes and degree of crystallinity (T_c) can be evaluated from the DSC data. Samples for DSC measurements were prepared from several tiny rectangular pieces cut from the polymer film to a mass ranging from 1.98 -24.24 mg for PSPE and 7.08 – 19.82 mg for HSPE. The temperature range studied was -40 to 200 °C. The heating/cooling rate was 10 °C/min.

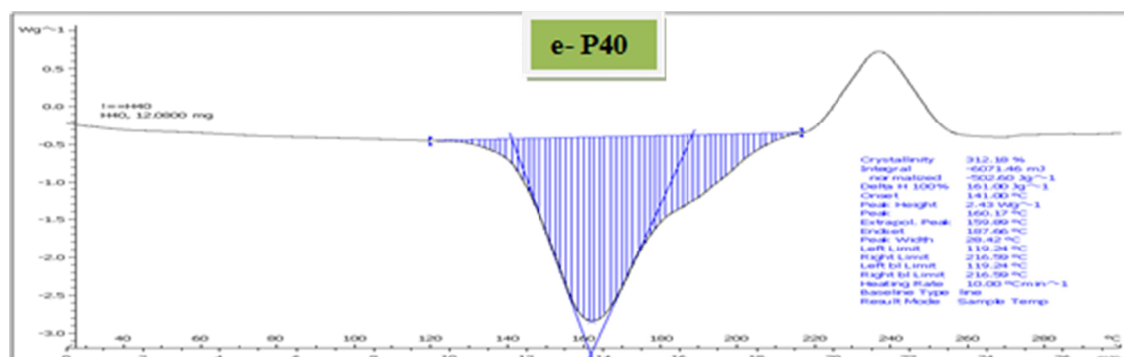
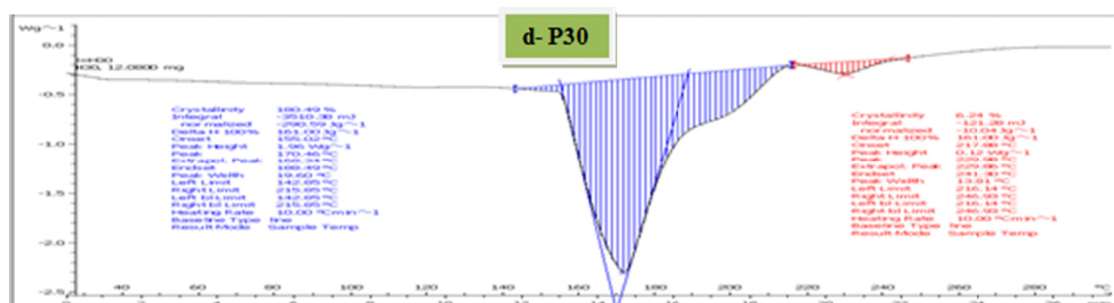
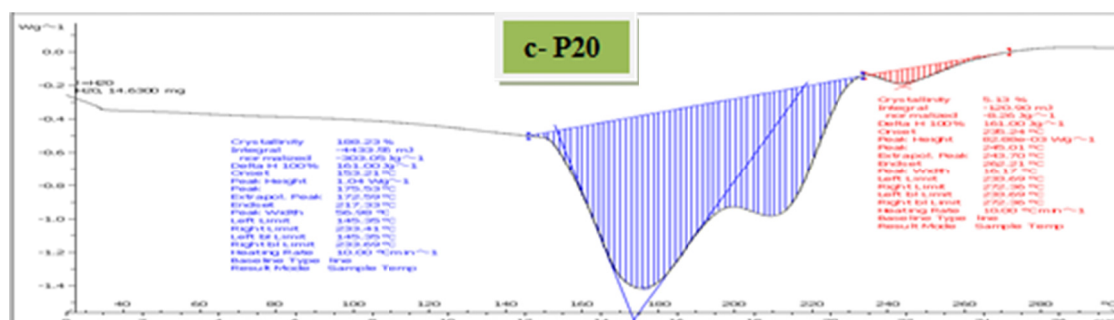
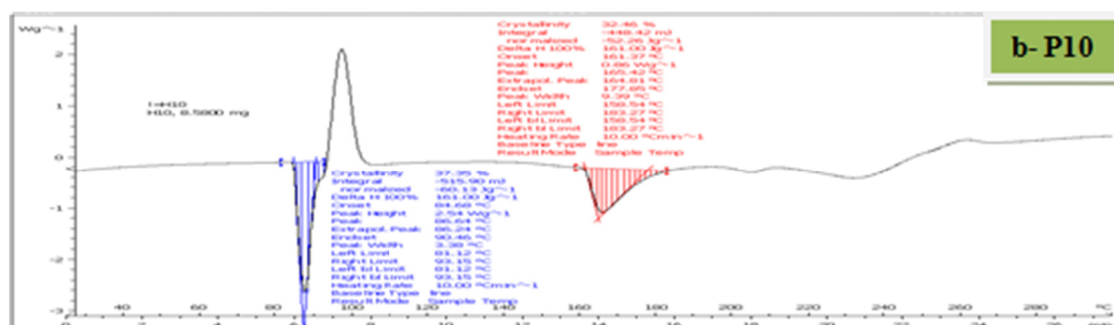
Table 3. Parameters for the Thermometric Analysis in PSPE

Coding	PVA: $_3\text{PO}_4$	Mass (g)	Temperature (°C)		
			T_g	T_m	T_c
P0	100:0	1.98	98.11	223.56	152.00
P10	90:10	4.48	80.78	221.06	166.27
P20	80:20	7.41	127.31	207.26	186.45
P30	70:30	8.27	117.63	156.46	-
P40	60:40	15.12	116.89	155.00	-
P50	50:50	13.53	101.13	164.59	160.05
P60	40:60	19.48	63.28	160.00	150.00
P70	30:70	24.40	79.41	129.09	79.41

Figure 4 shows the DSC analysis result of PSPE containing 0 wt.% (a); 10 wt.% (b); 20 wt.% (c); 30 wt.% (d); 40 wt.% (e); 50 wt.% (f); 60 wt.% (g); 70 wt.%. From the Figure and Table 3 it can be seen that, the first three samples show a very good T_m with an endothermic peak of slightly above 200 °C (i.e. 233.56, 221 and 207.26 for P0, P10 and P20 respectively) which indicates the huge percentage weight of PVA is present here. Noting the theoretical value of the thermal transition of the pure PVA as 230 °C, this result is closely related to that, and was in agreement with that of (Jelinska et al., 2010; Agrawal, 2004; Othman et al., 2011). However, probably due to the presence of chemical content, or intermolecular interaction (Mudigoudra et al., 2012) form among PVA and H_3PO_4 , the thermal transition starts to decrease with the increase in temperature.

The higher the melting point temperature the more the stability of the molecules therein, as such, pure PVA has more stable molecules than PVA/ H_3PO_4 composite. So, it can be seen that the T_m and T_c decreases (or even diminishes) as the concentration of the acid increase. This is a known fact that, in homogenized blends, the melting shifts to the lower level (Othman et al., 2011; Guirguis & Moselhey, 2012.). The other unusual humps cited in especially P0 and P60 could be as a result of the presence of the moisture inside the samples. Also the theoretical glass transition of the PVA alone is about 85 °C while the composite shows down and upwards shifts of this temperature. This could not be unconnected with the gradual increment in the concentration of the acid.





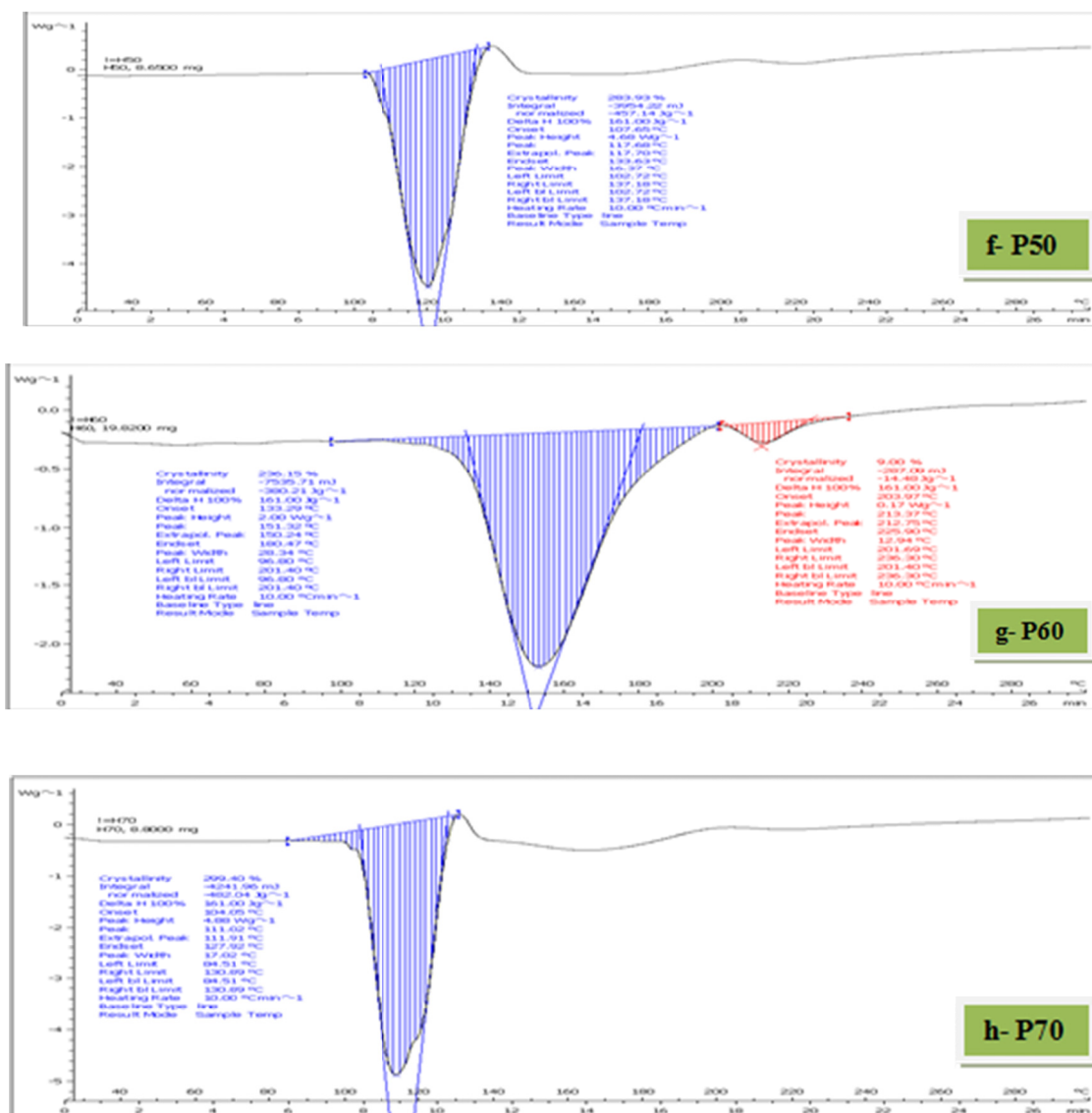


Figure 4. DSC Analysis of Solid (S) Solid Polymer electrolyte containing 0 wt.% (a); 10 wt.% (b); 20 wt.% (c); 30 wt.% (d); 40 wt.% (e); 50 wt.% (f); 60 wt.% (g); 70 wt.% (h)

4. Conclusion

In this study, we have introduced the noble of the pure solid polymer electrolyte at room temperature used as an electrolyte as well as a separator from the piece of PVA and H_3PO_4 such that, the PVA samples were kept constant while the phosphoric acid was varied at 0, 10, 20, 30, 40, 50, 60, and 70 wt. %. The PSPE results in higher conductivity of $2.56 \times 10^{-3} \text{ Scm}^{-1}$, at the compositions of 70 wt. %, in addition to that, we also observed that, the bulk modulus R_b decreases with the increase in concentration, recording 2.5Ω at the highest composition of 70 wt. %. The XRD analysis reveals the semi-crystalline peak of the PVA at $\theta=20^\circ$ border humps of the left indicating the inclusion of the acid and the crystallinity decreases as the acid concentration increases. This result was confirmed by the DSC analysis.

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