AC Conductivity and Conduction Mechanism of Iron (II) Chloride (FeCl_2) /Poly (vinyl alcohol) (PVA)/Poly (vinylpyrrolidone) (PVP) Polymer Blend Electrolyte

Huda Alfannakh (✉ halfannakh@kfu.edu.sa)

King Faisal University <https://orcid.org/0000-0001-9626-2950>

Original Research

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Abstract

Blend sample based on Poly (vinyl alcohol) (PVA)/Poly (vinylpyrrolidone) (PVP), (50:50), and their blend electrolyte with different weigh ratio of Iron (II) Chloride (FeCl_2), (0.5, 1 and 2 wt.%), have been prepared using casting method. The AC electrical properties of these samples were investigated in the frequency range 1 kHz to 1 MHz and in temperature ranging from 30 to 150 °C. The real and imaginary impedance, frequency and temperature dependence, indicate the enhancement of the electrical conductivity of the blend due to addition of FeCl_2 . Pure and electrolyte blend samples were found to be characterized by a plateau region at low frequency and high temperature, and this plateau region increases with increase in temperature and /or FeCl_2 weight ratio. *Nyquist* plots for pure and blend electrolyte samples presented depressed semicircles at all temperatures. For pure blend sample, the ac conductivity was attributed to hopping mechanism while for blend electrolyte sample ionic conduction represents the predominant conduction mechanism. *Jonscher* universal power law was used to study the conduction mechanism for pure and electrolyte blend sample. The activation energy for blend and blend electrolyte samples was calculated and two activation regions were appeared for blend electrolyte samples.

Introduction

The characteristics of PVP depend on its molecular weight. PVP is available in several grads (molecular weights) and it is soluble in aqueous organic solvent in addition to its nontoxic character, all these characteristics give PVP great flexibility for several applications. PVP was used as adhesive agent for improving the strength of paper in paper manufacture and also in painting industry and ink, imaging, lithography and other industries[1, 2]. In medical applications[3, 4], PVP has been used as a blood plasma expander for trauma victims[5]. It can also be used as a binder in many pharmaceutical industries[6, 7], as it is easily absorbed into the body, making it gain great importance in the field of pharmacology. PVP also was used as a pore forming agent to enhance the polymeric water filter membrane[8] and its addition can affect the quality of the resulting ultrafiltration polymeric membrane.

Poly(vinyl alcohol) (PVA) represent one of the famous polymer which has a wide range of applications such as packing, biomedical and medical applications [9–11]. It has some natural characterization such as non-toxicity, chemical resistance biocompatibility and hydrophilicity. Beside these characteristics, PVA is a good member for preparation of several kinds of polymer blends and polymer electrolytes. PVA is water soluble polymer and it was used in several kind of water filtration researches[12, 13]. Both polymers PVA and PVP has excellent wetting properties and readily forms films, which makes it good as a coating or an additive to coatings.

The field of polymer blend and polymer blend composite represent one of the advanced area for research in polymer physics and this due to its contribution in several types of applications [14]. Two or more polymers can be used to prepare a new blending of polymeric material with unique and interesting properties, different from those in the original components [15] .

Polymer blend of synthetic polymers like PVP and PVA is very important for various applications, such as, electrochemical devices[16], heat transfer [17], wound healing[18–20], tissue engineering[21, 22], biological medical applications[3, 4], artificial skin[22] and composite membranes[23, 24].

Since PVP and PVA possess an interesting set of dipolar groups[25], the formation of polymer blend electrolyte based on such polymer blend will be interesting for the scientists and researchers. The dielectric relaxation spectroscopy (DRS) may be a suitable method for investigating the properties of such electrolytic polymer blends in a wide range of frequency and temperature [26]. Dielectric relaxation spectroscopy is a useful technique to investigate the relaxation behavior of polymeric electrolyte.

The present work is aimed to investigate the AC conductivity for the prepared polymeric blend electrolyte composed of PVA/PVP blend loaded with different weight ratio of FeCl_2 .

2. Experimental

2.1 Materials

PVA (86.7–88.7 mol% hydrolysis, Mw 67000) and PVP (Mw 55 000 and Mw 360 000) were supplied by Sigma Aldrich (USA). FeCl_2 (98 %) was supplied by Sigma Aldrich (China). PVA, PVP, and FeCl_2 were used without further purification or treatment. De-ionized water was used as the solvent for preparing the polymer solutions.

2.2 Preparation Of Polymer Blend Film Composite

An aqueous PVA solution (10 wt.%) and PVP (10 wt.%) was prepared by adding the polymer powder to De-ionized water, followed by heating the solution under magnetic stirring at 80 °C for 2 hours. Both polymeric solution was mixed together and left on magnetic stirrer for overnight to form a homogenous PVA/PVP polymer blend. Iron (II) chloride (FeCl_2) with different weight was dissolved in 5 ml of De-ionized water and ultrasonic homogenizer (Hielscher homogenizer 400W) for 5 min. PVA/PVP/ FeCl_2 solutions (different weight percent of FeCl_2) were made by mixing the polymer blend and FeCl_2 solutions under ultrasonicated for 30 min at room temperature. Films were obtained using casting technique using a Petri dish and after drying at room temperature (32 °C) for 3 days. The polymer blend composite PVA/PVP/ FeCl_2 with different FeCl_2 weight percent (0.5, 1, 2 and 3 wt. %) were kept in glass desiccator with silica gel.

2.3 Composite Characterization

The polymer blend composite samples were characterized by FT-IR, and AC spectroscopy. FTIR spectra for PVA/PVP blend and FeCl_2 electrolyte samples were recorded using Perkin Elmer spectrum 400. Dielectric measurements were carried out using Keithley Instruments 4200 Semiconductor Characterization System over a frequency range 1 kHz to 1 MHz and temperature range from room temperature up to 150 °C.

3. Results And Discussion

3.1. FTIR Characterization

The effect of FeCl_2 on the polymer blend was investigated and analyzed by using FTIR spectroscopy. The FTIR spectra of pure blend polymer (50% PVA: 50% PVP) and the blend polymer doped with different weight ratio of FeCl_2 (0.5, 1, 2 and 3wt. %) is shown in Figure 1. The transmittance peaks observed in the pure blend 50% PVA: 50% PVP are 3313 cm^{-1} , 2922 cm^{-1} , 1743 cm^{-1} , 1654 cm^{-1} , 1272 cm^{-1} , 846 cm^{-1} . These peaks are attributed to O-H stretching, CH_2 asymmetric stretching, C=O stretching, C=C stretching, C-O-C stretching, CH_2 bending respectively [27, 28].

The spectrum of FeCl_2 /PVA/PVP showed peaks at a similar wavenumber to that of the polymer blend PVA/PVP, suggesting that FeCl_2 did not affect the structural characteristics of blend. However, a change in the relative intensity of certain bands (2922 cm^{-1}) in the spectrum of the 1wt.% FeCl_2 sample. This peak is become sharper and a clear peak at 2853 cm^{-1} was obtained. This can be attributed to the hydrogen bonding between the blend and FeCl_2 (see Figure 2).

3.2 Impedance Spectroscopy

One of the most important techniques for detecting the dielectric relaxation processes in polymer, polymer blend and polymer composite is the impedance spectroscopy. The analysis of complex impedance represents a powerful tool to investigate and distinguish between different components which may contribute in the material. From the complex impedance analysis and the expected equivalent circuit based on its data, we can get more deep information about the physical process that takes place inside the material under test. Figures 3(a & b), 4(a & b), 5(a & b) and 6(a & b) show the frequency dependence of the real and imaginary parts (Z' and Z'') of impedance on at different temperatures ranging from 30 to 140 °C for PVA/PVP blend and doped samples. It is clear from the figure that both real and imaginary parts decrease with increasing temperature.

Pure blend sample (PVA/PVP) does not show any significant dependence on temperature for either the behavior of the real part (Z') or the imaginary part (Z'') of impedance. This behavior can be ascribed to the dielectric nature of the blend sample, and thus the behavior is closer to the capacitive behavior over the used frequency range. The motion of the main chain due to thermal activation is small and it adds a slight change in the impedance behavior.

It is clear that, blend electrolyte samples have different behavior than pure sample. At low temperatures, the behavior has a tendency to that of the capacitive reactance and the relationship between impedance and frequency is linear relation. This can be attributed to the incapability of the dipoles to oriented or respond to the applied frequency. The frequency-independent plateau region increases with temperature and it is connected with the dc-conductivity of the polymer blend electrolytes (see Figures 4(a), 5(a) and 6(a)). In the high frequency region, the capacitive component of real part of impedance

was predominating and the dependence become linear. As the concentration of FeCl_2 increases, the high frequency dispersion (Z'') is shifted towards the higher frequency side, and the conductivity was found to be increased (see Figures 4(b), 5(b) and 6(b)). The shift of dispersion to high frequency indicates the decrease of relaxation time which attributed to increase in electrical conductivity of samples.

Nyquist plots for pure and blend electrolyte samples with different weight ratios (0.5, 1 and 2 wt.%) of FeCl_2 are illustrated in figures 7, 8 (a & b), 9 (a & b) and 10 (a & b) respectively. The plot exhibits depressed semicircles for all samples and at all temperatures. Pure samples did not show a complete semicircle shape due to the insulating nature of the blend and the inability of the charge carriers to move except at high temperature. This may be attributed to the chain's entanglement. For electrolyte blend and for temperature higher than 50 °C, the semicircles were followed by spikes at lower frequency side which increase with increasing salt weight ratio and temperature. These results indicates a non-Debye relaxation nature (a single relaxation time) for pure and electrolyte blend samples [29]. The equivalent circuit which represents such depressed semicircle plot comprising a constant phase element capacitor (CPE) in parallel with a resistor. This combination was connected in series with another constant phase capacitive element to represent the shape of the spike at low frequency region [30].

Figure 11 shows the effect of FeCl_2 weight ratio (concentration) and temperature on Nyquist plots. It was noted that increasing the temperature and /or FeCl_2 weight ratio reduces the diameter of the semicircle, which indicates the active participation of the ionic conductivity through the samples.

3.2 AC conductivity

Figures 12(a), 13(a), 14(a) and 15(a) show the variation of AC electrical conductivity (σ_{ac}) as function of frequency and at different temperatures for pure 50wt% PVA-50wt%PVP blend and doped with 0.5, 1 and 2 wt.% FeCl_2 respectively. The AC conductivity (σ_{ac}) is found to be temperature and frequency dependent. AC conductivity behavior of all blend electrolyte samples is characterized by plateau region (frequency-independent) at higher temperature. This plateau become more predominated for higher weight ration doped samples (2 wt.%).

For low frequency region, σ_{ac} for blend electrolyte samples was frequency independent especially at temperature higher than 50 °C. This can be interpreted due to the ionic conduction which dominates the ac-conductivity that arises as a result of the response of the polymer chains and dipoles to applied frequencies. For pure blend sample, it was notice that the dependence of the ac-conductivity on frequency continue up to 80 or 90 °C, and the ac-conductivity increases due to hopping mechanism which increase with frequency increases [31, 32].

Figures 12(b), 13(b), 14(b) and 15(b) shows the variation of $\log(\sigma_{ac})$ with $1000/T$ for pure blend, 0.5, 1 and 2 wt.% FeCl_2 electrolyte blend samples respectively. It is obvious from these set of figures that the ac-conductivity was thermally activated and the mobility of charge carrier and charge density increase with

temperature increases. The main chain and segmental chain motion play an important role for the conductivity behavior for pure and electrolyte samples [32].

The addition of electrolyte to polymer or polymer blend can form complexes with the polymeric chains, which impede the movement of the chains and makes them require higher energy to contribute to the ac-electrical conductivity, and the ionic conductivity has the advantage in the active participation of the ac-electrical conductivity. Occasionally, at small concentrations of salt, the fluid can act as a plasticizer and facilitate the movement of the polymeric chains. From the table it was note that the electrolyte blend samples have two linear regions, the first at low temperatures in which the contribution return to polymer with activation energy in the range 0.13 to 0.19 eV (greater than pure blend) which indicates a complex formation between some chains and the ionic liquid. The activation energy for second region (ionic conduction contribution) was ranging between 0.2 to 0.43 eV. The activation energy was decrease with increasing of salt concentration.

Table 1 Values of the activation energy for pure and electrolyte PVA/PVP blend sample.

Sample	Activation Energy (eV)		R ²
	Region I	Region II	
PVA/PVP	0.12		0.985
PVA/PVP/0.5wt% FeCl2	0.402	0.192	0.991 & 0.998
PVA/PVP/2wt% FeCl2	0.432	0.183	0.992 &0.996
PVA/PVP/1wt% FeCl2	0.277	0.132	0.922 & 0.942

Jonscher universal power law can be used to analyses the overall behavior of AC conductivity based on the following equation [33, 34]:

$$\sigma(\omega) = \sigma_{dc} + A \omega^n$$

where $\sigma(\omega)$, σ_{dc} , A, ω and n are defined as the total conductivity, dc-conductivity, constant, angular frequency and an exponent factor. The dependence of $\log \sigma$ vs. $\log f$ in the high temperature region and the dependence of exponent n on temperature are illustrated in Figures 16 and 17 for pure blend and blend electrolyte samples respectively. The exponent values n was determined by knowing the slope of $\log \sigma$ vs. $\log f$ relation and are summarized in tables 2 to 5.

It was noticed that the values of the exponent n varies with salt concentration [35, 36]. Also, pure sample has two regions follows the power law which are depend on the frequency range. The change of n with concentrations was attributed to the change of the interface between different constituents of the samples. In the case of polymer blend electrolyte, the host was a polymeric blend beside a reminder of the individual polymers (partial miscibility) which were not contributed in the blend process. At this

hypothetical region, one can imagine that three contributors are present, the blend electrolyte, the electrolyte solution (reminder) and the immiscible reminder of PVA and PVP polymers electrolytes.

It was also noted that the behavior of the exponent n with the temperature for pure blend sample is linear whether at temperatures below or higher than glass transition point. At higher temperatures (see Figure (16-b)), two linear regions were detected and n was linearly depend on temperature. Whereas, for electrolyte samples the behavior of $n(T)$ follows the exponential function (n decreases exponentially with temperature) [37]. This means that the movement of chains and side chain may be activated due to addition of electrolyte (FeCl_2). This will excites the movement of ions significantly and increase the conductivity reducing the effect of interfacial regions which increase the probability of hopping mechanism for ions [38]. Also, according to Correlated barrier hopping model (CBH), as temperature increase the ionic hopping between sites to overcome the potential barrier will increase [37, 39, 40].

The following tables (Table 2 to 5) illustrates the values of the exponent n , the coefficient A , and the dc-conductivity (σ_{dc}) for pure sample at different temperatures. It is obvious that the increase in the dc-conductivity was accompanied by a decrease in the value of n and thus the expansion of the plateau region at the expense of the exponential region of $\sigma(f)$ [36].

These results confirm the role of thermal activation of the main chains beside the side chains which are contribute significantly to the electrical conductivity. It is clear that the role of chain, movement due to thermal activation, was greater than the role of the applied alternating voltage. The presence of two regions for pure blend sample, (Figure (16)) at high temperatures, may be attributed to the presence of more than one conduction mechanism beside the possibility of contribution of polymer reminders side by side to polymer blend.

Table 2 Values of the exponent n , coefficient A and the dc conductivity for pure PVA/PVP blend sample.

T	Low Temperature					
	n	A	σ_{dc}			
30	1.112	2.67E-06	2.67E-06			
40	1.021	7.38E-06	7.38E-06			
50	1.054	8.85E-06	4.21E-06			
60	1.043	9.77E-06	4.57E-06			
70	0.975	2.14E-05	5.54E-06			
80	0.974	3.86E-05	3.86E-06			
Region I			Region II			
	N	A	σ_{dc}	n	A	σ_{dc}
90	0.822	9.13E-06	1.37E-04	0.97	2.76E-05	7.64E-06
100	0.893	7.20E-06	1.02E-04	0.97	2.76E-05	9.05E-06
110	0.891	9.31E-06	1.00E-04	0.97	2.76E-05	1.03E-05
120	0.595	5.69E-05	5.69E-04	0.89	4.90E-05	1.83E-05
130	0.523	1.04E-04	7.77E-04	0.87	6.16E-05	2.31E-05
140	0.433	2.11E-04	1.43E-03	0.82	9.00E-05	3.37E-05
150	0.361	2.90E-04	2.75E-03	0.78	1.26E-04	4.72E-05
160	0.272	5.53E-04	5.24E-03	0.71	2.08E-04	7.80E-05

Table 3 Values of the exponent n, coefficient A and the dc conductivity for pure 0.5 wt.% FeCl₂ PVA/PVP electrolyte blend sample.

T	n	A	σ_{dc}
30C	1.030	3.52E-06	3.52E-05
40C	0.878	5.02E-06	3.52E-05
50C	0.747	7.09E-06	3.52E-05
60C	0.543	5.38E-06	2.42E-04
70C	0.400	2.94E-06	2.47E-04
80C	0.276	2.16E-03	1.62E-03
90C	0.177	6.35E-03	4.76E-03
100C	0.124	1.15E-02	8.62E-03
110C	0.093	1.79E-02	1.34E-02
120C	0.077	2.52E-02	1.89E-02
130C	0.062	3.07E-02	2.30E-02
140C	0.046	4.78E-02	3.59E-02

Table 4 Values of the exponent n, coefficient A and the dc conductivity for pure 1 wt.% FeCl₂ PVA/PVP electrolyte blend sample.

T	n	A	σ_{dc}
30C	0.522	1.19E-03	4.48E-04
40C	0.427	1.09E-02	4.07E-03
50C	0.245	1.19E-02	4.44E-03
60C	0.227	1.32E-02	4.95E-03
70C	0.165	2.22E-02	8.30E-03
80C	0.082	7.37E-02	2.76E-02
90C	0.048	9.25E-02	3.46E-02
100C	0.022	1.17E-01	4.39E-02
110C	0.012	1.42E-01	5.30E-02
120C	0.008	1.57E-01	5.87E-02
130C	0.003	1.56E-01	5.86E-02
140C	0.002	1.59E-01	5.94E-02

Table 5 Values of the exponent n, coefficient A and the dc conductivity for pure 2 wt.% FeCl₂ PVA/PVP electrolyte blend sample.

T	n	A	σ_{dc}
30C	0.759	1.35E-04	2.25E-05
40C	0.594	6.02E-04	1.00E-04
50C	0.464	1.91E-03	3.19E-04
60C	0.338	5.72E-03	9.53E-04
70C	0.202	1.70E-02	2.83E-03
80C	0.158	2.49E-02	4.14E-03
90C	0.126	3.52E-02	5.87E-03
100C	0.087	5.33E-02	8.88E-03
110C	0.063	7.55E-02	1.26E-02
120C	0.049	9.72E-02	1.62E-02
130C	0.041	1.15E-01	1.91E-02
140C	0.046	1.24E-01	2.07E-02

Conclusion

This work investigated the study of some electrical properties of a polymeric blend composed of PVA and PVP as well as a polymer blend electrolyte by adding FeCl₂ with different weight ratios (0.5, 1 and 2 wt.%). Impedance analysis was carried out in the frequency range from 1 kHz to 1 MHz under different temperatures (30 to 150°C). The incorporation of FeCl₂ in the blend matrix enhanced the electrical conductivity of the blend and the ac-conductivity increase as the FeCl₂ weight ratio increases. Nyquist plots for pure and blend electrolyte samples presented depressed semicircles at all temperatures. Pure samples did not show a complete semicircle shape due to the insulating nature of the blend and the inability of the charge carriers to move except at high temperature.

Nyquist plots diameter reduced as the temperature and /or FeCl₂ weight ratio increases which indicates the active participation of the ionic conductivity through the samples. For pure blend sample, the AC conductivity was attributed to hopping mechanism. The results for blend electrolyte samples was interpreted based on the increase of chain mobility due to thermal activation and the contribution of ionic conduction. Jonscher universal power law was used to study the conduction mechanism for pure and electrolyte blend sample. The activation energy for blend and blend electrolyte samples was calculated and two activation regions were appeared for blend electrolyte samples.

Declarations

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Conflict of interest

The authors declared that there is no conflict of interest.

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Figures

Figure 1

FTIR pattern for 50%PVA: 50%PVP with (a) 0%, (b) 0.5%, (c) 2% and (d) 3% wt.% FeCl₂

Figure 2

(a) PVA/PVP blend and (b) 2% FeCl₂ blend electrolyte sample

Figure 3

a) real part of impedance (Z') and (b) Imaginary part of impedance (Z'') for PVA/PVP polymer blend.

Figure 4

a) real part of impedance (Z') and (b) Imaginary part of impedance (Z'') for 0.5wt% FeCl₂/PVA/PVP polymer blend electrolyte.

Figure 5

a) real part of impedance (Z') and (b) Imaginary part of impedance (Z'') for 1wt% $\text{FeCl}_2/\text{PVA}/\text{PVP}$ polymer blend electrolyte.

Figure 6

a) real part of impedance (Z') and (b) Imaginary part of impedance (Z'') for 2wt% FeCl₂/PVA/PVP polymer blend electrolyte.

Figure 7

Complex plane plots (Z' - Z'') for PVA/PVP polymer blend at various temperatures

Figure 8

Complex plane plots ($Z'-Z''$) for 0.5 wt.% $\text{FeCl}_2/\text{PVA}/\text{PVP}$ blend composite at various temperatures

Figure 9

Complex plane plots ($Z'-Z''$) for 1 wt.% $\text{FeCl}_2/\text{PVA}/\text{PVP}$ blend composite at various temperatures

Figure 10

Complex plane plots ($Z'-Z''$) for 2 wt.% $\text{FeCl}_2/\text{PVA}/\text{PVP}$ blend composite at various temperatures

Figure 11

Complex plane plots (Z' vs. Z'') for PVA/PVP/ FeCl_2 blend electrolyte samples (a) at 80C (b) at 110°C

Figure 12

(a) Frequency dependence of AC conductivity at different temperatures (b) $\log(\sigma)$ versus $1000/T$, for pure PVA/PVP blend sample.

Figure 13

(a) Frequency dependence of AC conductivity at different temperatures (b) $\log(\sigma)$ versus $1000/T$, for 0.5 wt.% FeCl₂ PVA/PVP electrolyte blend.

Figure 14

(a) Frequency dependence of AC conductivity at different temperatures (b) $\log(\sigma)$ versus $1000/T$, for 2 wt.% FeCl₂ PVA/PVP electrolyte blend.

Figure 15

(a) Frequency dependence of AC conductivity at different temperatures (b) $\log(\sigma)$ versus $1000/T$, for 1 wt.% FeCl₂ PVA/PVP electrolyte blend.

Figure 16

(a) $\log \sigma$ vs. $\log f$ in the high-frequency region and (b) variation of the exponent n against the temperature for PVA/PVP blend.

Figure 17

$\log \sigma$ vs. $\log f$ in the high-frequency region and variation of the exponent n against the temperature (a, b) 0.5 wt.% (c & d) 1.0 wt.% (e & f) 2.0 wt.% FeCl₂ PVA/PVP blend electrolyte.