

EFFECT OF NANO ZnO CONCENTRATION ON STRUCTURE AND ELECTRICAL CONDUCTION IN PEO - LiClO₄ BASED COMPOSITE POLYMER ELECTROLYTE

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(Received: 15 / 6 /2011)

ABSTRACT

Nano ZnO with an average size of 8 nm was prepared by thermal decomposition of zinc oxalate at 450 °C. PEO - LiClO₄ based composite polymer electrolyte was prepared by using solution cast technique. The samples were characterized by X-ray diffraction (XRD), differential scanning calorimetry (DSC) and infra red (FT-IR) techniques. Effect of nano ZnO ceramic filler concentration on the structure of composite and its electrical properties (DC-conductivity , AC- conductivity , dielectric constant , dielectric loss and impedance) at different frequencies and temperatures was studied. Melting temperature (T_m) of PEO decreased with the addition of LiClO₄ salt and nano ZnO filler due to increasing the amorphous state of polymer. All composite samples showed an ionic conductivity with highest value for the sample containing 0.5 mole of nano ZnO. All the results are correlated and discussed.

Keywords: PEO-based polymer electrolyte; Ionic conductivity; Nano composites

INTRODUCTION

There has been increasing interest in the development of solid polymer electrolytes, due to their applications in solid-state electrochemical device, and particularly in solid state rechargeable lithium batteries [J.M.Tarascon *et al.*, (2001)] Solid polymer electrolytes for lithium batteries have many advantages over their counterpart liquid electrolytes, such as processing flexibility, but their conductivity at room temperature is usually too low to be applicable. Improvement the ionic conductivity of these electrolytes can be achieved by fillers. At least there are two types of fillers to improve

the ionic conductivity of PEO – based polymer electrolytes. One is small organic molecules, such as ethylene carbonate (EC) and propylene carbonate (PC) [A.G.Bishop et al., (1996)], which can enhance the polymer electrolyte conductivity to a moderate value. But this makes the systems appearing as a gel or like a liquid, which loses the advantages of solid polymer electrolytes to some extent. The other is dry inorganic compounds as ceramic powders which can enhance not only the conductivity but also the stability of the polymer in the meantime [E. Quartarone et al., (1998)]. This paper is a part of our program to prepare polymer composite electrolytes and study the effect of nano materials on their physical properties. In this paper, nano ZnO with an average particle size of 8 nm was synthesized using thermal decomposition method and then used to prepare polymer nano composite electrolytes $(\text{ZnO})_x(\text{PEO})_{12.5-x}(\text{LiClO}_4)$ with different concentrations of ZnO ($x = \text{mole}$, in the range of 0 – 1.75) as a ceramic filler. The results showed an ionic conductivity of the polymer electrolyte with a highest value of $6.33 \times 10^{-5} \text{ ohm}^{-1} \cdot \text{cm}^{-1}$, at room temperature, for the sample containing 0.5 mole ZnO.

EXPERIMENTAL

Preparation of nano ZnO filler

Pure zinc acetate [$\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$, LOBA Chemie PVT.LTD.] and oxalic acid [$\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$, Bio – Chem.] were used as raw materials. 300 ml of 0.15 M oxalic acid solution was added slowly (drop by drop) to 300 ml of 0.1 M zinc acetate solution under stirring for 18 hr. A white precipitate of zinc-oxalate was obtained, which was filtered and washed with acetone several times to remove impurities. The ZnO powder was then dried at 120 °C for 30 minutes and finally heated for 30 minutes at 450 °C.

Preparation of polymer nano composite electrolytes

PEO (with an average molecular weight of 8×10^6 , Segma - Aldrich), previously prepared nano ZnO with a mean particle size of 8 nm, and LiClO_4 (Segma – Aldrich) were used as starting materials to prepare polymer nano composite electrolytes. The composite samples were synthesized by the conventional solution cast technique. PEO and LiClO_4 were dissolved in acetonitrile and magnetically stirred to get a homogeneous solution. An appropriate weight of nano zinc oxide was then added to the solution and stirred for 6 hrs to get a white viscous solution with high homogeneity. After that, the viscous solution was poured on petter dish and left to dry at room temperature for two days to allow the solvent to be evaporated slowly. All the films obtained with different concentrations of filler were then dried under vacuum for 10 hrs. and kept in a desiccator.

Characterization of samples

X-ray diffraction were performed on the investigated samples using a Philips X-ray diffractometer (Model PW 1710) with $\text{CuK}\alpha$ radiation ($\lambda = 1.54 \text{ \AA}$) in the range of $2\theta = 4 - 60^\circ$. Thermal analysis was performed on the samples investigated in air atmosphere and at a temperature range of 298 – 353 K using differential scanning calorimetry

technique (DSC) by Shimadzu DSC-60H. FT-IR spectra of the samples were recorded in the range of 400 - 3000 cm^{-1} using KBr pellet technique on IR-Brucker, Vector 22, Germany.

RESULTS AND DISCUSSION

The XRD pattern of zinc oxide is shown in Fig.(1). It shows typical peak patterns characterized for ZnO with the hexagonal wurtzite - structured (p63mc) with high crystallinity as indexed in the standard data (JCPDS card No. 36 - 1451, $a = 3.249 \text{ \AA}$ and $c = 5.206 \text{ \AA}$) [J.Liu et al., (2005)]. XRD line broadening was used to estimate the grain size of the powders according to Scherrer formula [H.P.Klug et al., (1970)]

$$D = 0.9 \lambda / \beta \cos \theta \quad (1)$$

Where D is the average diameter of the grains, λ is the wavelength of the Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$), θ is the Bragg angle and β is the full width half maximum in radians calculated using Gaussian fitting. The results showed an average crystalline size of about 8 nm. XRD of pure PEO and composites of $(\text{ZnO})_x(\text{PEO})_{12.5-x}(\text{LiClO}_4)$, ($x = 0, 0.5, 0.75, 1.25, \text{ and } 1.75$ mole) are also shown in Fig. (1). The XRD of PEO polymer showed crystallinity behaviour due to the appearance of characteristic diffraction peaks in the range of $2\theta = 19 - 23^\circ$ [B. Wunderlich et al., (1980)].

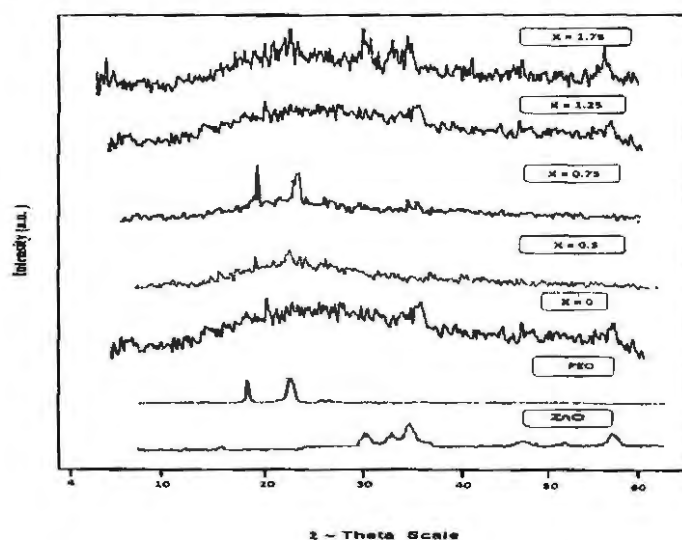


Fig. (1): XRD. Patterns of ZnO , pure PEO and $(\text{ZnO})_x(\text{PEO})_{12.5-x}(\text{LiClO}_4)$, ($X = 0, 0.5, 0.75, 1.25, 1.75$ mole).

Whereas, the XRD of the composites containing LiClO_4 and nano ZnO showed a remarkable reduction in the intensity of XRD peaks of pure PEO, specially for the sample containing 0.5 mole ZnO, which showed a complete amorphous nature. This can be attributed to a destruction effect of the filler on the ordered arrangement of the polymer side chains, and as a result, an enhancement in the amorphous phase.

DSC thermograms of pure PEO and $(\text{ZnO})_x(\text{PEO})_{12.5-x}(\text{LiClO}_4)$, ($x = 0, 0.5, 0.75, 1.25, \text{ and } 1.75$ mole) were displayed in Fig. (2). The relative percentage of crystallinity (X_c) has been calculated by taking into account pure 100 % crystallinity for PEO [B. Wunderlich et al., (1980)] and using the equation

$$X_c = \Delta H_c / \Delta H_p \quad (2)$$

Where, ΔH_p equals to 203 J / g which is the heat enthalpy of 100 % crystalline PEO [B. Wunderlich et al., (1980)], and ΔH_c is the heat enthalpy of composites. The calculated relative crystallinity X_c and the data obtained from DSC thermograms are summarized in Table (1). From which it can be seen that the melting temperature (T_m) of crystalline PEO phase decreases with increasing the amount of ZnO. This refers to increase the flexibility with increasing the amount of ZnO in the PEO matrix.

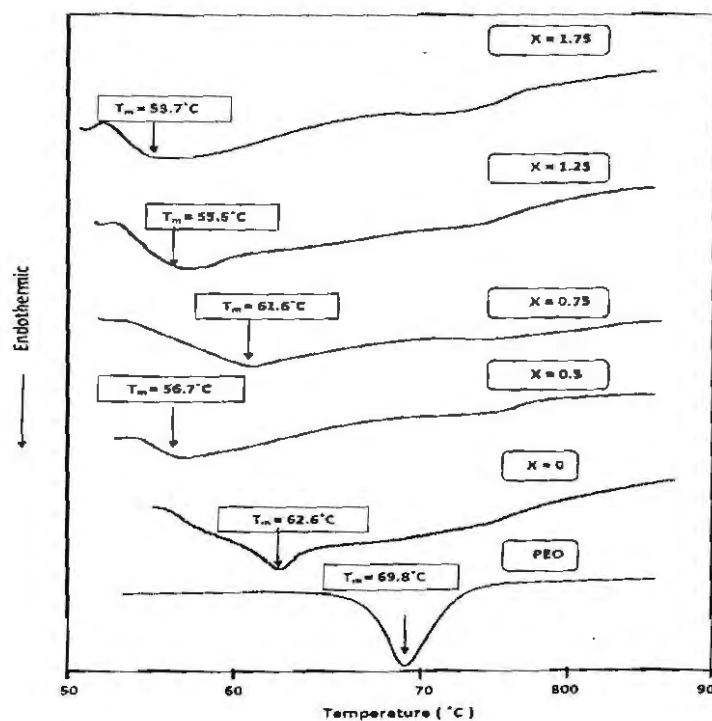


Fig. (2): DSC curves of pure PEO and $(\text{ZnO})_x(\text{PEO})_{12.5-x}(\text{LiClO}_4)$, ($X = 0, 0.5, 0.75, 1.25, 1.75$ mole).

Table (1): Melting temperatures (T_m), melting enthalpy (ΔH_m) and crystallinity (X_c) for pure PEO and $(ZnO)_x(PEO)_{12.5-x}(LiClO_4)$.

(X), (mole)	T_m ($^{\circ}C$)	ΔH_m (J/g)	X_c (%)
pure PEO	69.8	139.1	68.5
0	62.6	52.3	25.8
0.5	56.7	71.3	35.1
0.75	61.6	160.6	79.1
1.25	55.5	94.8	46.7
1.75	53.7	197.1	97.1

The IR spectra of ZnO, PEO, $LiClO_4$ and $(ZnO)_x(PEO)_{12.5-x}(LiClO_4)$, ($x = 0, 0.5, 0.75, 1.25, \text{ and } 1.75$ mole) are shown in Fig. (3). The spectra of the composite systems display some spectral features that are similar to those of pure PEO. However, the incorporation of both $LiClO_4$ and nano ZnO particles leads to the shift of some bands of PEO

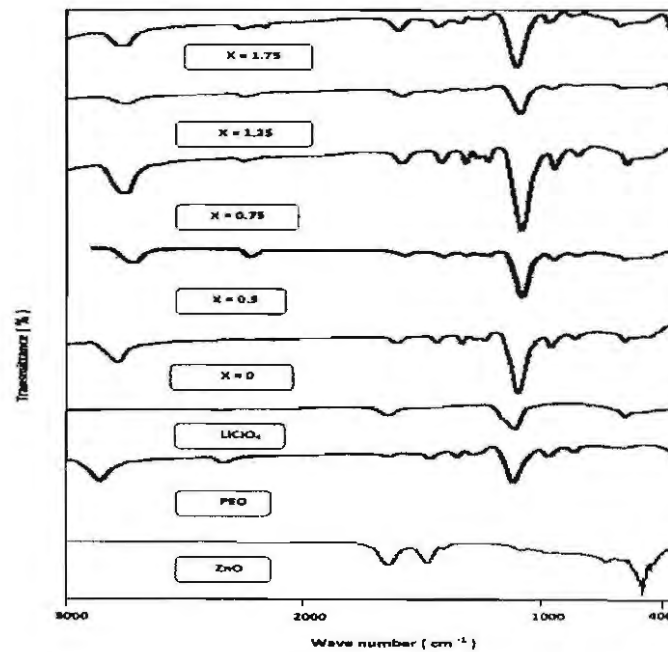


Fig. (3): IR Patterns of ZnO, PEO, $LiClO_4$ and $(ZnO)_x(PEO)_{12.5-x}(LiClO_4)$, ($X = 0, 0.5, 0.75, 1.25, 1.75$ mole).

polymer. At frequency range ($1050 - 1160 \text{ cm}^{-1}$), significant changes were observed in the width and intensity of the vibrational bands of PEO due to the addition of LiClO_4 salt and nano ZnO filler. These changes were attributed to the effect of both LiClO_4 and nano ZnO on C - O - C symmetric vibrational modes. Metal oxygen stretching frequency of 525 cm^{-1} in pure ZnO is shifted to 587 cm^{-1} in the composite indicating the Vander-Waals interaction between ZnO and PEO polymer. Moreover, the shifts occurring in the vibrational band of C - O - C at $800 - 1000 \text{ cm}^{-1}$ refer to the electrostatic interaction of Zn^{2+} metal ion with the ether oxygen [B.L. Papke et al., (1982)] and [S.J. wen et al., (1996)]. Generally, the changes occurring in IR spectra confirm the interaction process in the polymer nano composite electrolytes samples with each of Zn^{2+} and Li^+ ions. This interaction may be associated with the interaction of Zn^{2+} ion and oxygen atom in PEO macromolecule. Because Zn is a transition metal, Zn^{2+} has intense tendency to form coordination compound with oxygen atom in PEO macromolecule. This interaction may weaken the bond strengths of C-O-C in PEO macromolecule.

DC - Electrical conductivity

The temperature dependence of electrical conductivity (DC) of $(\text{ZnO})_x(\text{PEO})_{12.5-x}(\text{LiClO}_4)$, ($x = 0, 0.5, 0.75, 1.25, \text{ and } 1.75$ mole) is investigated in a temperature range of $293 - 323 \text{ K}$ and illustrated in Fig. (4). The overall feature of the Arrhenius plots is almost similar for all investigated composites. The conductivity data are summarized and given in Table (2). Generally, it can be seen that the addition of nano ZnO in the PEO - LiClO_4 matrix causes a high enhancement in the conductivity of the composite electrolyte. Electrical conductivity reached a value higher 100 times more than that of the polymer electrolyte system PEO + LiClO_4 , Table (2). The increase in conductivity may be attributed to an increase occurring in the flexibility of the composite by introducing ZnO into PEO matrix, due to the high interface area between the matrix and the dispersed nano ZnO particles [L. fan et al., (2003)], [J.Maier (1995)] and [W.Wieczorek et al., (1998)]. Moreover, the increase in amorphous phase within the PEO-matrix due to the addition of nano ZnO causes an enhancement in the segmental motion of the polymer chains and hence increases the conductivity.

However, the increase in conductivity of the composite with increasing the concentration of nano ZnO filler is not linear, Fig. (5). The two maxima appearing in Fig. (5) have been also reported by few workers for other systems of ion conducting gel polymer electrolyte composite [J.P.Sharma et al., (2007)] and also for solvent free composite polymer electrolytes [B.K. Choi et al., (1996)], [S.A. Hashmi et al., (2000)] and [G.P.Pandey et al., (2008)]. The first conductivity maximum is possibly due to the dissociation of ion aggregates salt into the free ions with the addition of nano sized ZnO particles, whereas the second conductivity maximum is related to the composite effect and explained on the basis of formation of a conducting interfacial space - charge double layer between the nano sized ZnO particles and polymer electrolytes [J.Maier (1995)] and [B.Kumar (2004)]. The decrease in conductivity after the second

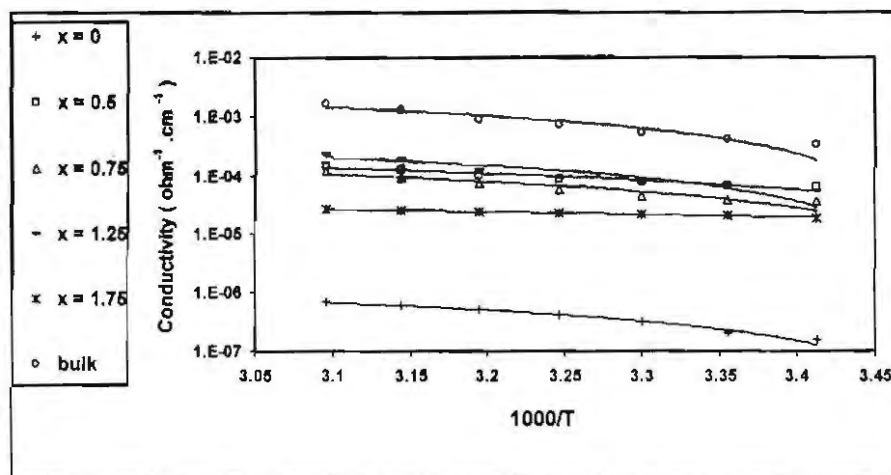


Fig. (4): Temperature dependence of both DC conductivity for composite samples with different X values and bulk at X=0.5 mole.

Table (2): DC and bulk conductivity (σ_{dc}, σ_b) data for pure PEO and $(ZnO)_x (PEO)_{12.5-x} (LiClO_4)$ at room temperature (293 K).

(X), (mole)	(E_a)(eV)	$\sigma_{dc} (ohm^{-1}.cm^{-1})$	$\sigma_b (ohm^{-1}.cm^{-1})$	(E_a)(eV)
Pure PEO	0.40	1.25×10^{-9}	7.00×10^{-7}	0.33
0	0.46	1.55×10^{-7}	1.60×10^{-4}	0.55
0.5	0.24	6.33×10^{-5}	3.42×10^{-4}	0.43
0.75	0.36	3.72×10^{-5}	1.10×10^{-4}	0.60
1.25	0.41	5.43×10^{-5}	2.00×10^{-4}	0.53
1.75	0.55	1.95×10^{-5}	5.51×10^{-5}	0.62

conductivity maximum Fig.(5) is generally related to the blocking effect of filler particles, which hinders the motion of mobile ions [B.Kumar et al., (2006)]. The conductivity results, Table (2), showed a highest conductivity value for the sample containing 0.5 mole ZnO because this sample containing the highest amorphous structure (show XRD) which facilitate the transport of ionic charge carriers , as will be seen later.

The increase observed in conductivity with temperature, Fig. (4), can be attributed to the decrease in the viscosity and hence the flexibility chain of the sample [K.Tsunemi et al., (1983)].

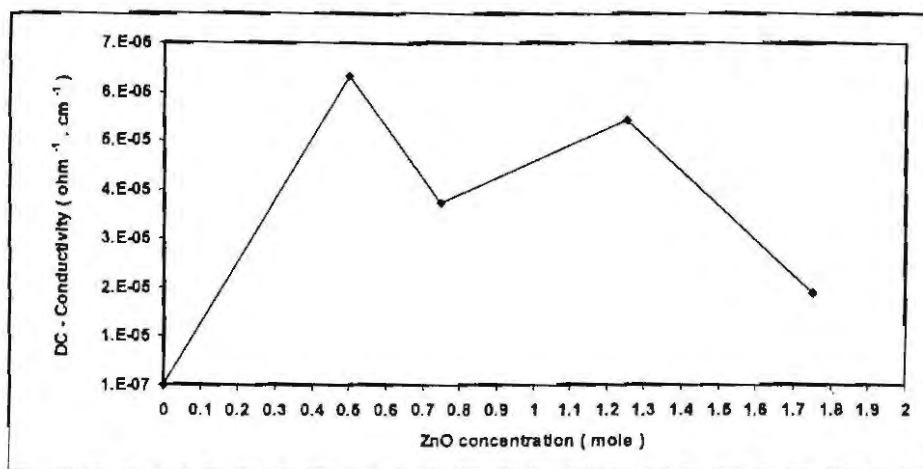


Fig. (5): ZnO concentration dependence of dc –conductivity (σ_{dc}) for $(\text{ZnO})_x(\text{PEO})_{12.5-x}(\text{LiClO}_4)$, ($X = 0, 0.5, 0.75, 1.25, 1.75$ mole) at room temperature (293 K).

The conductivity data present in **Table (2)** show an activation energy value (0.46 eV) for the sample free from nano ZnO filler more than that of the samples containing ZnO filler (0.24 eV). This low value of activation energy in our sample can be attributed to the amorphous nature of the sample that will facilitate the Li^+ ion motion in the polymer network [R.Baskaran et al., (2007)], as shown in the results of impedance. The electrical devices that work over a wide temperature range should have uniform conductivity, thus, materials with low activation energy is a good electrical system [J.M.G. Cowie et al., (1998)]. Therefore, the composite system containing 0.5 mole nano ZnO was chosen to be more studied using AC – conductivity, dielectric behaviour, and impedance character.

Impedance spectroscopy analysis

Impedance plots (Z' Vs. Z'') in the complex plane for $(\text{ZnO})_{0.5}(\text{PEO})_{12}(\text{LiClO}_4)$ at different temperatures showed similar behavior. A typical plot is shown in **Fig. (6)**, which demonstrates a depressed semicircular portion followed by a spike. This spike refers to ionic conductivity and is characteristic of a blocking double layer capacitance whose magnitude can be estimated from any position on the spike using the equation. $Z'' = 1 / 2\pi f C_{dl}$, where f is the frequency and C_{dl} is the capacitance at the frequency f . The ionic conductivity of the solid polymer nano composite electrolyte $(\text{ZnO})_{0.5}(\text{PEO})_{12}(\text{LiClO}_4)$ was derived from the ac impedance analysis. The high depression of semicircular portion at high frequencies, in complex impedance, is a result of the increase in ionic conductivity with increasing the frequency [L. fan et al., (2003)]. The equivalent circuit of the polymer nano composite electrolyte system is determined from the complex impedance spectrum and given in **Fig. (6)**, where, R_1 is the bulk resistance of the electrolyte, C_1 is the bulk capacity of the electrolyte and C_2 is a capacity of bulk electrode – electrolyte interface. The bulk ionic conductivity of $3.42 \times 10^{-4} \text{ ohm}^{-1} \cdot \text{cm}^{-1}$

at room temperature was calculated using the equation $\sigma_b = L / R_b A$, where L is the thickness of the polymer nano composite electrolyte film and A is the surface area of the film. The resistance of the electrolyte (R_b) was determined from the intercept of the impedance spectrum on the Z' real axis. The temperature dependence of the bulk conductivity showed Arrhenius behaviour, Fig. (4), as that reported by dc - conductivity. The bulk conductivity data are also reported in Table (2). The difference between dc and impedance data is attributed to the grain boundaries present between the particles.

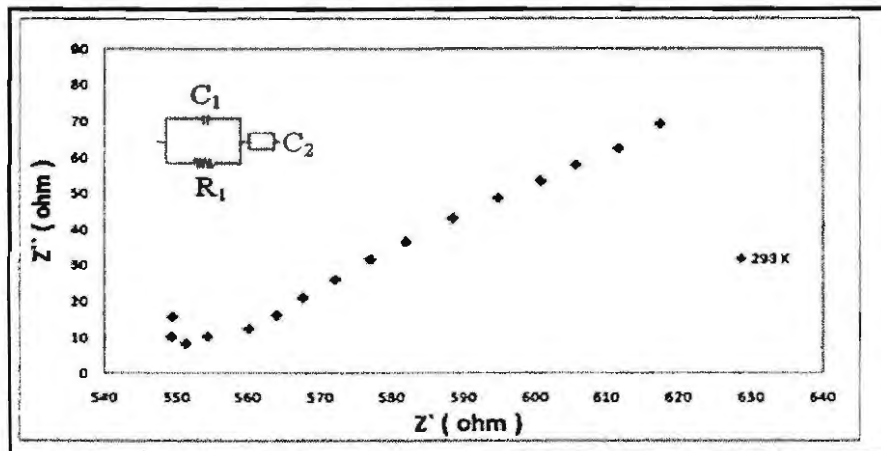


Fig. (6): Plot of Z'' versus Z' for $(ZnO)_{0.5}(PEO)_{12}(LiClO_4)$, at room temperature (293 K).

AC - conductivity

In order to give information on the type of polarization present in $(ZnO)_{0.5}(PEO)_{12}(LiClO_4)$ sample, the ac-electrical conductivity (σ_{ac}), at temperatures between 293-323 K and at frequency range of $10^2 - 10^5$ Hz was studied, Fig. (7). It can be seen that, the conductivity increases with each of the frequency and temperature. The frequency dependence of conductivity is attributed to the mobility of charge carriers. The increase in σ_{ac} with temperature can be explained on the basis that raising the temperature causes more structure relaxation and releasing more of Li^+ ions attached oxygen of PEO to become more mobilized. This may be also due to increasing the drift mobility and hopping frequency of charge carriers.

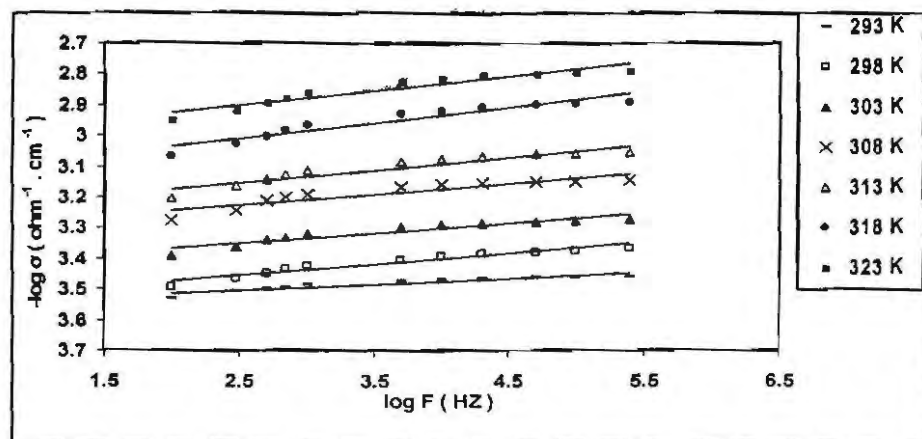


Fig. (7): Variation of electrical conductivity (AC) with frequency at different temperatures for $(\text{ZnO})_{0.5}(\text{PEO})_{12}(\text{LiClO}_4)$.

Dielectric permittivity and loss studies

The frequency dependence of dielectric constant ϵ' at different temperatures for $(\text{ZnO})_{0.5}(\text{PEO})_{12}(\text{LiClO}_4)$ sample is shown in Fig. (8.a). It can be seen that ϵ' decreases as frequency increases. This decrease is relatively sharp at lower frequencies and slower at higher ones. The decrease in dielectric permittivity with increasing frequency can be associated to the inability of dipoles to rotate rapidly leading to a lag between frequency of oscillating dipole and that of applied field [Arvind awadhia et al., (2006)]. The dielectric permittivity obtained in our system ($\epsilon' = 8.58 \times 10^5$, at room temperature) is higher than that for other systems of polymer electrolytes (ϵ' lie in the range of $9.8 \times 10^3 - 1.52 \times 10^5$, at room temperature). Such high dielectric constant values in our sample can be attributed to high ionic conductivity on account of the presence of nano particles of zinc oxide and its good distribution within the matrix of poly ethylene oxide and also due to the good interactions between the lewis acidic sites on the surface of those particles and the ions of the lithium perchlorate [C.A. Finch (1973)]. When the temperature is raised, dielectric constant also enhances due to the facilitation in orientation of dipoles within the matrix of PEO.

The temperature dependence of dielectric constant at different frequencies is shown in Fig. (8.b). The variation of dielectric constant with temperature can be divided into two ranges, in the low temperatures range (from 293 to 303 K), the change in the dielectric constant is weakly dependent on the temperature but in the higher temperatures range (from 303 K to 323 K), the dielectric constant is strongly dependent on the temperature. The increase of dielectric constant with temperature is generally attributed to two mechanisms. The first is to the decrease of viscosity of the polymer nano composite electrolyte [K.P. Singh et al., (1992)] and the second is to the dissolving of any small concentration of crystalline and semi-crystalline phases into the amorphous phase [Arvind awadhia et al., (2006)]. This in turn influences the polymer dynamics and thus the dielectric behavior. The present of the two ranges in Fig. (8.b) may be attributed to the effect of the two mechanisms. Whereas, at lower

temperature range the viscosity is low and crystalline phases are present, thus the dielectric behavior is weakly dependent on the temperature, while at higher temperatures the opposite trend is predominate.

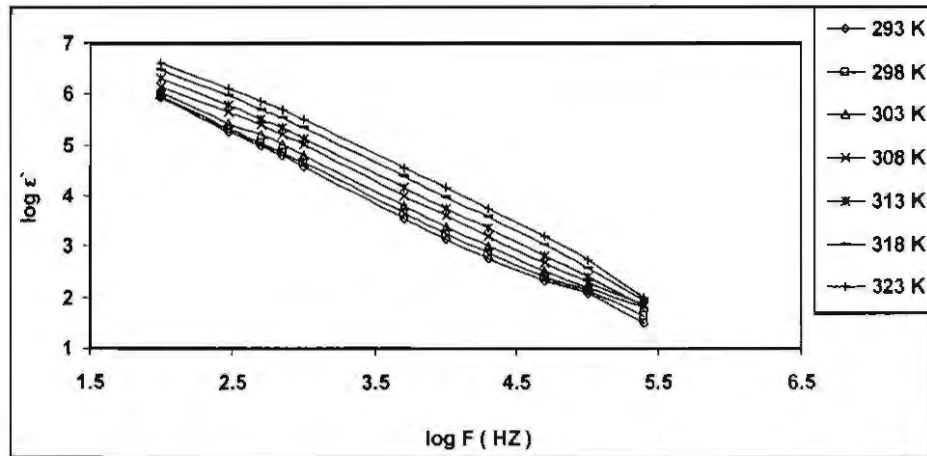


Fig. (8,a):Variation of dielectric constant with frequency at different temperatures frequencies for $(ZnO)_{0.5}(PEO)_{12}(LiClO_4)$.

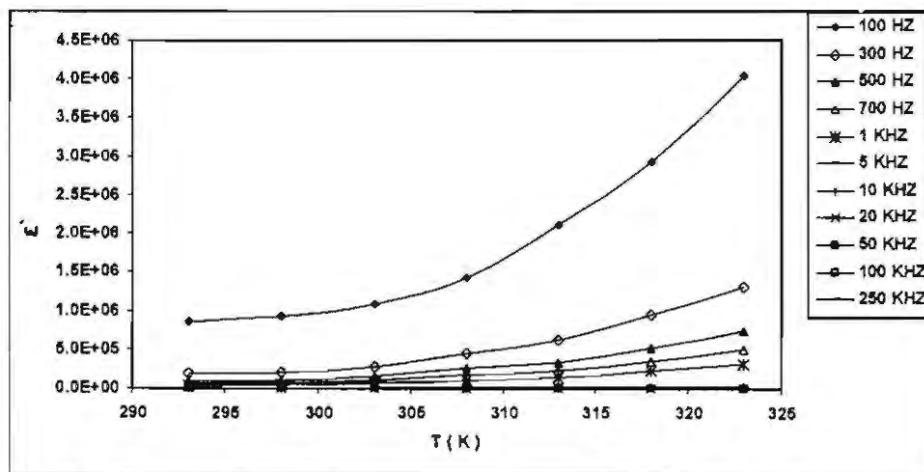


Fig. (8,b):Variation of dielectric constant with temperature at different frequencies for $(ZnO)_{0.5}(PEO)_{12}(LiClO_4)$.

Fig. (9 a,b) shows the variation of dielectric loss (ϵ'') with frequency and temperature for $(ZnO)_{0.5}(PEO)_{12}(LiClO_4)$ sample. The behaviours observed are similar to that found for dielectric constant. Where the dielectric loss decreased with increasing the frequency due to high periodic reversal of the field at the interface, the contribution of charge carriers (ions) towards the dielectric loss decreases with increasing frequency and also this decreasing can be attributed to the reduction of the diffusion of the ions in the polymer matrix with increasing the frequency. Also the increase in dielectric loss with increasing temperature can be attributed to the relaxation of the dipoles in co-operation with the resulting drop in the relaxation time.

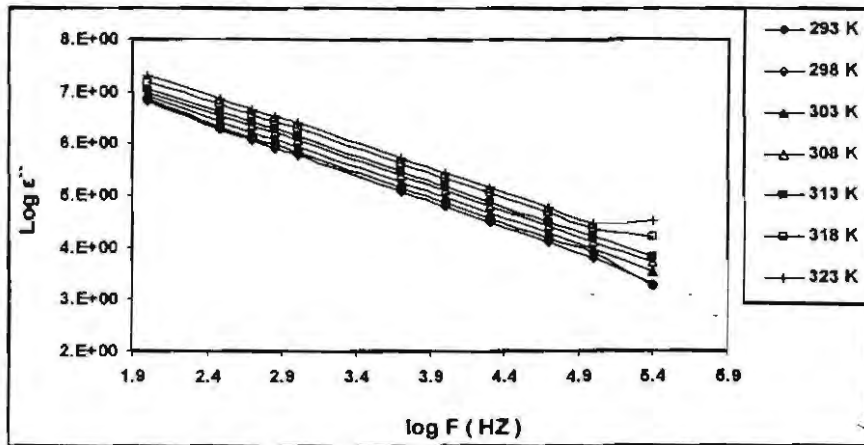


Fig. (9,a): Variation of dielectric loss with frequency at different temperatures for $(ZnO)_{0.5}(PEO)_{12}(LiClO_4)$.

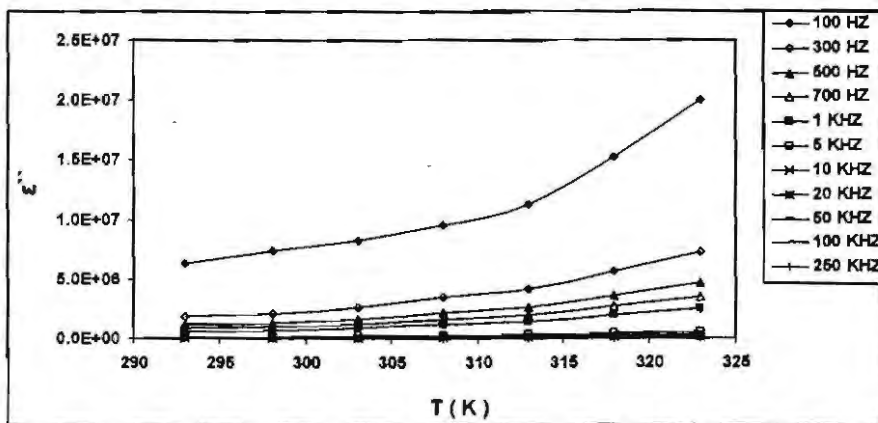


Fig. (9,b): Variation of dielectric loss with temperature at different frequencies for $(ZnO)_{0.5}(PEO)_{12}(LiClO_4)$.

CONCLUSIONS

Nano sized ZnO filler with an average particle size of 8 nm was prepared by a thermal decomposition of zinc oxalate. $(\text{ZnO})_x(\text{PEO})_{12.5-x}(\text{LiClO}_4)$ composite electrolytes containing nano sized ZnO particles with concentration of $x = 0.5, 0.75, 1.25$ and 1.75 mole have been synthesized. The samples were characterized by DSC, X-ray and Infra-red spectra. The shifts, broadening and reduction in the intensity of the IR-bands confirm the dissolution of the metal salt in the polymer matrix. The DSC analysis revealed to a change in the crystallinity and melting points of the composite samples with the different concentrations of the filler. All composite samples showed ionic conductivity, in which the addition of nano ZnO particles enhances the ionic conductivity of the based polymer by hundred times at room temperature. The sample containing 0.5 mole nano ZnO showed a conductivity of $6.33 \times 10^{-5} \text{ ohm}^{-1} \cdot \text{cm}^{-1}$ at room temperature, which is the highest conductivity value for all investigated samples. The electrical properties of this sample showed dielectric permittivity and loss values of 8.58×10^5 and 6.30×10^6 respectively at room temperature. These high values of DC-conductivity, dielectric permittivity and dielectric loss at room temperature lead to make the sample as a promising material for lithium battery as an application in the solid state electrochemical devices.

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الملخص العربي

تأثير تركيز نانو أكسيد الزنك على التركيب والتوصيل الكهربى لمتراكب الكتروليتى بوليمري
مكون من بولي اكسيد ايثيلين وبيركلورات الليثيوم

ا.د / عبد الحميد البليهي و ا.د / وفاء عبدالله بيومي و عماد مسعود و ا.د / محمود أحمد موسى
قسم الكيمياء - كلية العلوم - جامعة بنها

تم دراسة تأثير اضافة تركيزات مختلفه من نانو أكسيد الزنك المحضر بحجم ٨ نانوميتر باستخدام طريقة التكمير الحراري لأكمالات الزنك عند درجة ٤٥٠ سيليزيه على التركيب والخواص الكهربيه لمتراكب بولي اكسيد ايثيلين - بيركلورات الليثيوم وكذلك تم التعرف على تركيب العينات تحت الدراسه باستخدام مجموعه مختلفه من التقنيات مثل الأشعه السينيه وتحت الحمراء وتحليل السعر الحراري التفاضلي وأظهرت النتائج ان درجة حرارة الذوبان للمتراب تقل مع اضافة كل من بيركلورات الليثيوم وأكسيد الزنك وبعد دراسة العينات المحضره وجد ان العينه التي تحتوي على تركيز ٠,٥ مول نانو أكسيد الزنك هي الأعلى في التوصيل الأيوني مقارنة بالعينات الأخرى .

