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Study of the electrical conductivity of poly (ethylene oxide) in different electrolytes

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Abstract

The electric conductivity of systems without and based on poly(ethylene oxide) polymer of seven binary solutions (KCl, NaCl, NaOH, LiCl LiBr, LiI and $NaNO_2$*-*H_2O*) and ternary solutions based on halogenated lithium (LiCl, LiBr and LiI-PEO-* H_2O*) was considered as function of changes in concentration and temperature.*

The experimental results revealed that the electric conductivity of solutions is influenced by the concentration, temperature and the characteristics of the solute such as the anionic radius, mobilitiy and diffusion coeffient. At 18 ° C, the maximum electrical conductivity was 7.01 S/m for an aqueous solution of poly(ethylene oxide) PEO / Lithium iodide (LiI at 0.1M) / water. In addition, the electric conductivity decreased in the order: PEO / (LiI) / H_2O*> PEO / (LiBr) / H2O> PEO / LiCl -* H_2O *under the same conditions of concentration and temperature.*

The activation energies of the ternary and binary systems are determined by the Vogel-Tamman equation -Fulcher (VTF). Therefore, the solution of the ternary LiI-PEO-H_2O *system shows good potential as working solutions for Lithium ion batteries considering its electrochemical and thermodynamic proprieties.*

Keywords: Poly (ethylene oxide) PEO; Electrolytes; Conductivity; Lithium ion battery; activation energy

1. Introduction

Polymer electrolytes have received remarkable research investments over the past decades due to the extreme demand for practical material application in devices, they provide an alternative approach to replace electrolytes based on body fluids. Lithium-ion batteries are suitable as energy storage / conversion devices (ECSD) due to the increasing demand in daily life. But certain safety problems concern them such as their poor thermal stability, flammable reaction products and electrolyte leaks and internal short circuits [1]. Although polymers are insulators, research work on the poly (ethylene oxide) (PEO) system and its alkali metal salts (NaI) has confirmed the non-negligible conductivity in polymers and which has reinforced the attention towards the development of polymer electrolytes. Their technological importance and was introduced in the form of solid polymer electrolytes due to their flexibility and deformability [2].

Polymeric electrolytes based on poly (ethylene oxide) with high molecular weight are one of the most promising candidates for the preparation of polymer electrolyte due to the flexibility of the polymer chain, the semi crystalline nature and the presence of an ether group at both sides, leads a rapid charge transfer (CT) ensuring interesting transport properties. Therefore, poly(ethylene oxide) based solid polymer electrolyte (SPE) is the most preferred host polymer in the research system due to its flexible backbone and its ability to solvate lithium ions with coordination number dependent on the salt concentration and anions characteristics. In addition, the main advantage of poly (ethylene oxide) is its elevated solvatation power. Therefore, it can easily form a complex with many alkali salts and provide a direct path for cation migration due to the presence of the $(–CH_2–CH_2–O–)_n$ group in the polymer backbone. Although, the low conductivity value (10^{-7}-10^{-6} S/cm) and the reduced mechanical properties of poly (ethylene oxide) at room temperature limit their use in devices. In order to overcome the disadvantage, the most familiar method is to change the morphology by incorporating: nanofiller, plasticizer, ionic liquid and salt with a large anion in the polymer matrix. [3].

Polymer combination is one of the best techniques adopted to advance the properties of the host polymer being a physical blend of two individual polymers. This gives in hand properties superior to those of the individual polymer and can be easily controlled by varying the composition of the polymer used [3-5].Further improvement in mechanical and electric properties can be achieve by adding both nano-charges and plasticizers. Another approach is to use organic solvents, but the resulting film suffers from the elevated instability and therefore the flammability of the solvent as well as its reaction with the lithium metal electrodes and limits its use as an efficient energy storage device. The ability to manage independently the physic-chemical properties and to improve the mechanical strength, ionic conductivity, voltage stability and thermal properties of an electrolyte is desirable in the development of electrochemical devices. Generally, in a mixture of polymers, the presence of two phases occurs, the first provides a conduction path to the ion and the latter provides mechanical stability to the electrolyte [5- 8].

Poly (ethylene oxide) (Fig.1) is an uncharged hydrophilic polymer which presents a very interesting model system for studying the influence of electrolyte solutions on electrical conductivity due to its complete miscibility with water in all proportions for a very wide range of degrees of polymerization[7,8].

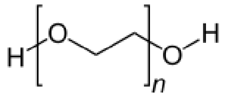

Figure1. Chemical structure of poly(ethylene oxide)[8].

This property is of great importance in elucidating the electronic properties of water and electrolyte near polymer chains induced by specific dipolar or hydrogen interactions, which can be reflected in changes in electrical conductivity.

The aim of this study consists of two parts: The first concerns the study of various parameters affecting the electric conductivity of a polymer solution ((poly (ethylene oxide) PEO)) in contact with various salts of alkali metals. Through, the choice of the best polymer/electrolyte system, the effect of the ionic radius, the ionic mobility and diffusion coefficient. In the second part we studied the systems of choice exhibiting the most advantageous electric conductivities in binary and ternary phase, the poly (ethylene oxide) in combination with lithium iodide (LiI) compared to the isolated lithium iodide in aqueous solution. Several parameters are clarified such as, the effects of the electrolyte and polymer concentration, temperature. Finally, the activation energies of the polymer / electrolyte and electrolyte system were determined using the Vogel-Tamman-Fulcher equation.

2. Materials and method

All chemicals were used as received. Pure deionized water (DI) was collected from a Millipore system.

The electric conductivity of a control sample comprising only dissolved Polyethylene (PE, commercial film) and Polyethylene oxide (PEO, 20,000 MW, 10 g in 15 mL heated in a water bath) is compared to that of different PEO based electrolytes containing either Lithium halides (LiCl, LiBr and LiI) and other salts Potassium Chloride (KCl), Sodium Chloride (NaCl), Sodium Hydroxide (NaOH) and Sodium Nitrate ($NaNO_2$) at a concentration from 0.01 to 0.1 M and a temperature of 18°C [9-13].

Once all components are dissolved, the resulting electrolyte is poured into a series of containing vessels having uniform diameters. A multimeter is used to monitor the conductivity and resistance of these solutions by placing the positive and negative electrodes at opposed ends of the containing vessel (Fig. 2) [9].

The solutions for the mobility and diffusion measurements were prepared in calibrated volumetric flasks using pure deionized water. The solutions were freshly prepared and de-aerated for about 30 min before each set of runs [14].

The mobility of ions is determined by analyzing the time interval between two steady states based on a simplified model. By altering the electric field, this approach is applicable to the measurement of the mobilities of different ion species [14-16, 18].

$$\mu^0 = \frac{Speed\ of\ ion\ (m\ s^{-1})}{Potential\ gradient(V\ m^{-1})}\ (m^2\,V^{-1}s^{-1}) \qquad (1)$$

The diffusion coefficients of ionic species have been obtained through conductometric measurements or ionic mobility. The expression relating diffusion coefficient (D) to its mobility at a temperature T is given as [15, 17-18].

$$D_i = \frac{k_B\,T}{|z_i|\,e} u_i^0 \ \ (\,m^2 s^{-1}) \qquad (2)$$

Where Z is charge (valence) of the ion, e is electronic charge , μ^0 is the mobility of the ionic species at infinite dilution, k_B is the Boltzmann constant.

A useful expression relating the diffusion coefficient D and the radius of a spherical diffusing species is given as Stokes Einstein equation [18],

$$D_i = \frac{k_B\,T}{6\pi\,\eta\,r_i} \ \ (m^2 s^{-1}) \qquad (3)$$

Where η is the viscosity of the solution (poise).

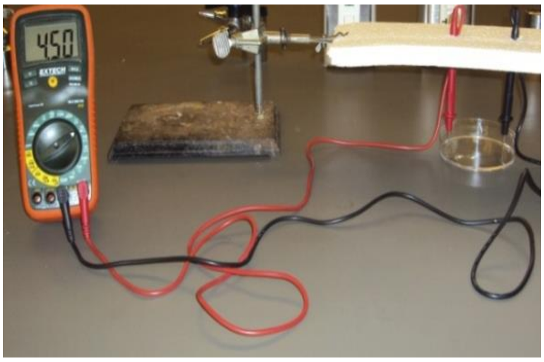

Figure 2. Experimental setup where the resistivity of a pure poly(ethylene oxide) -based electrolyte is measured [9].

The present formulas were used for determining resistivities and conductivities of the solutions [9]: The distance between electrodes was maintained at 5 cm.

Resistivity in Ω.m = (resistance in MΩ) × (distance between electrodes in cm) × (1 m/100 cm)

Conductivity in S/m = 1 /(resistivity in Ω.m)

Conductivity in µS/cm = (conductivity in S/m) × (1000000 µS/1S) × (1 m/100 cm)

3. Results and discussion

3.1. Choice of the effective electrolyte/polymer system

The choice of the best electrolyte/ polymer system to ensure the highly electric conductivity with great mobility requires the comparison of their conductivity properties in different electrolytes solutions. Fig.3a and Fig.3b show the electrical conductivities of the different electrolyte solutions based on poly(ethylene oxide) and polyethylene (PE).

It appears from the analysis of the Fig.3a and Fig.3b that the lithium salts (LiCl, LiBr and LiI) had very interesting electric conductivity values. While the system with sodium hydroxide (NaOH) gives intermediate conductivity when compared with all electrolytes. It is also interesting to note that the electric conductivity of the system with sodium chloride (PEO / NaCl) increases slightly compared to its analogue potassium chloride KCl. A cap on conductivity is recorded especially for the system with lithium iodide as electrolyte (PEO / LiI).

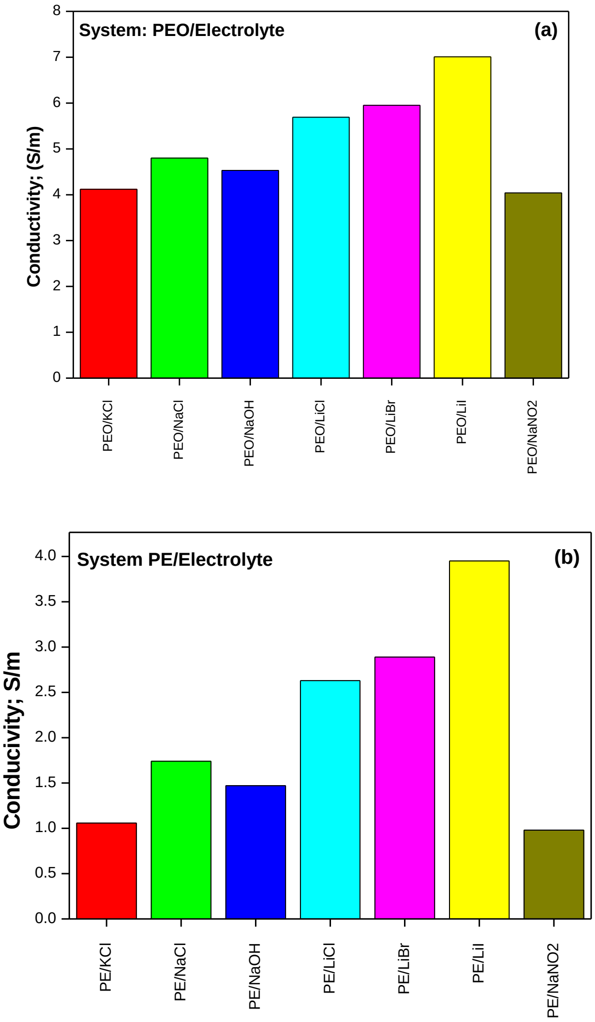

Figure 3. Comparison of the electrical conductivities for different systems: (a) poly (ethylene oxide)/salt) and (b) and polyethylene (PE/salt) at 18 ° C.

3.2. Effect of electrolyte parameters on the conductivity

3.2 .1. Effect of ionic radius

To assess the influence of the cationic radius on the conductivity of the poly(ethylene oxide)/electrolyte systems. A solutions of electrolytes based on poly (ethylene oxide) (PEO) (10 g in 15 mL of water) are prepared containing 0.1M NaCl, KCl and LiCl. The results are presented in Fig.4. Varying the cationic radii of dissolved salts can improve the conductivity of a polymer electrolyte solution. In the absence of any dissolved salt, the PEO electrolyte solution had a conductivity of 3.06 S m^{-1}. When the lithium halides salts (LiCl, LiBr and LiI) have been dissolved with the polymer water system, the conductivity is increased proportionally to the cationic radii. The larger the cation size becomes, the smaller the conductivity becomes, and the greater the resistance becomes. Also, a good linear relationship (R = 0.98) was found between the conductivity of the electrolyte/polymer systems and the ionic radii of the dissolved salts.

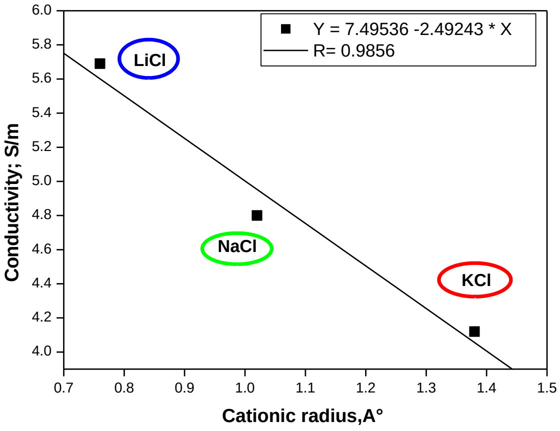

Figure 4. Change in electric conductivity of an aqueous electrolyte based on poly(ethylene oxide) containing 0.1 M of a series of lithium salts (LiCl, NaCl and KCl) as a function of cationic radius at 18 ° C.

The variation in ionic radii of anions leads to an increase in electrical conductivity with increasing anionic radii (Fig.5). An excellent linear relationship (R = 0.98) was found between the conductivity of the electrolyte/polymer systems and the anionic radii of the dissolved salts.

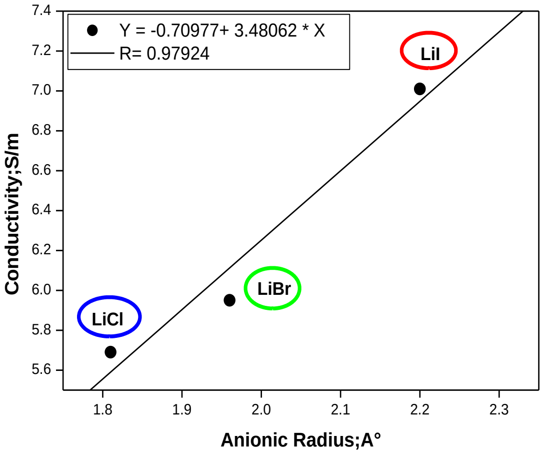

Figure 5. Variation of the electric conductivity of an aqueous electrolyte based on poly(ethylene oxide) containing 0.1 M of a series of lithium salts (LiCl, LiBr and LiI) as a function of the anionic radius at 18 ° C.

This observation can again be attributed to the dissociation constant of the ion pairs, which in this case increases with decreasing cation radius [8]. For the Cl$^-$ series, a smaller cation such as Li$^+$, with a large solvated radius [9] would interact more weakly with Cl$^-$ anions in solution compared to a larger cation such as K$^+$, which has a small solvated radius. Compared to the reverse trend mentioned above for the halide series, the ability observed for the alkali metal series supports the hypothesis that the ability of cations to couple with anions is a complex interaction that depends on several variables factors.

The solvatation of anions in protic solvents occurs through hydrogen bond interactions. Protic solvents, however, have poor electrochemical stability due to acidic protons (O− H). The dissolution of a lithium salt therefore occurs by the solvation of the Li$^+$ cations by the formation of coordination bonds between the Li$^+$ cations and the lone electron pairs of the solvent-donor atoms. Anions also form coordination bonds with Li$^+$ cations using the pairs of electrons isolated on the donor atoms (F, O, N, etc.).The competition between the solvent and the anions for the coordination of Li$^+$ cations determines the solvated species present in the electrolyte. Thus, ionic solvation (solvent interactions −Li$^+$ cation) and the ionic association tendency of anions (anion−Li$^+$ interactions) are important characteristics of electrolytes which are governed by the solvent / anion structure: steric factors that influence the formation of the coordination bond and compaction around the cation, the energy constant of the salt lattice, the polarizability of the ions, the charge delocalization and the solvatation medium.

3.2.2. Effect of cationic mobility

When the (LiCl, NaCl, KCl) salts were dissolved into the electrolyte/polymer systems, the conductivity increased in proportion with the ionic mobility of the cations for the dissolved salts (Fig.6).

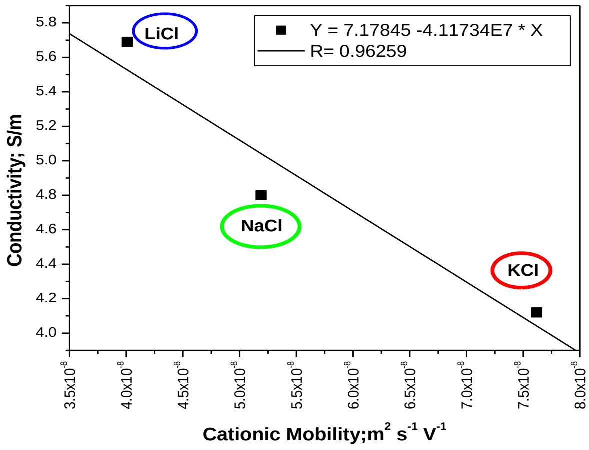

Figure 6. Variation of the electric conductivity of an aqueous electrolyte based on poly (ethylene oxide) containing 0.1 M of a series of lithium salts (LiCl, NaCl and KCl) as a function of cations mobility at 18 ° C.

3.2.2. Effect of ion mobility and diffusion coefficient on conductivity

The influence of anionic mobility and the electrolytes cation-diffusing power on the conductivity of poly(ethylene oxide) /electrolyte systems is presented in Fig.7.

Firstly, a similar trend was observed between the variation of anionic mobility and the diffusion coefficient. An inverse proportional relationship is observed for the anions, small values of diffusion coefficients and anionic mobilities produce high values of electrical conductivity. Our results are in agreement with those modelled by de Summerfield [19].

In an electrochemical cell, the anode and cathode are attached to opposite ends of the circuit with an electrolyte material separating them allowing ion transfer? When a lithium ion battery is discharged, negatively charged electrons flow from the anode to the cathode. At the same time, the positively charged Li$^+$ cations flow in the opposite direction. This concerted movement of charged species maintains overall charge neutrality while generating electricity. A high transport rate of Li$^+$ cation is necessary to achieve high battery power because the mobility of Li$^+$ cation in solution is often one of the main sources of battery impedance.

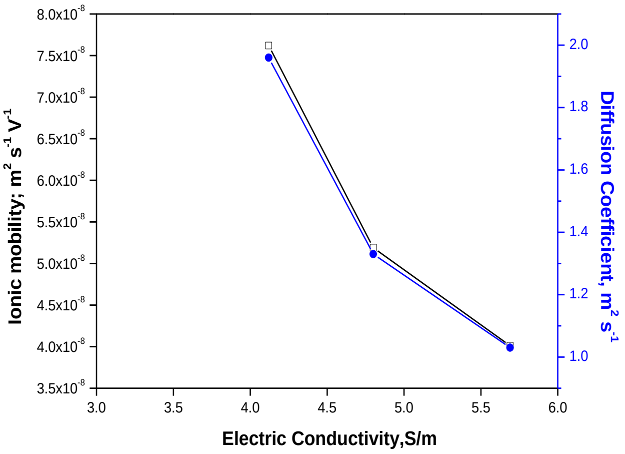

Figure 7. Variation of electric conductivity of poly(ethylene oxide)/LiCl, LiBr and LiI) systems as a function of anions mobilities and diffusion coefficients at 18 ° C.

3.3. Study of the poly (ethylene oxide) (PEO) / lithium iodide (LiI) system

3.3.1. Effect of electrolyte concentration

The variation of the specific (σ) and molar (Λ) conductivity for LiI and LiI /poly(ethylene oxide) PEO at 18 ° C as a function of lithium iodide (LiI) concentration are given in Table .1 and Fig.8.

In general, it is known that for strong electrolytes the variation of the concentration exerts a complex influence on the conductivity. The observation is that the conductivity increases with the number of charge carriers as well as the evolution of their nature. It can be seen that, for slightly concentrated solutions, this evolution is proportional.

Table 1: Variation of the specific (σ) and molar (Λ) conductivities for LiI and LiI/poly(ethylene oxide) at 18 ° C as a function of the lithium iodide (LiI) concentration

$\sqrt{C}$ mol/m^3	σ(PEO/LiI) S/m	Λ(PEO/LiI) S m^2 mol^{-1}	σ(LiI) S/m	Λ(LiI) S m^2 mol^{-1}
1.000	7.182	7.182	4.122	4.122
1.414	7.183	3.592	4.123	2.062
1.732	7.184	2.395	4.124	1.375
2.000	7.185	1.796	4.125	1.031
2.236	7.186	1.437	4.126	0.825
2.449	7.190	1.198	4.130	0.688
2.646	7.192	1.027	4.132	0.590
2.828	7.200	0.900	4.140	0.518
3.000	7.205	0.801	4.145	0.461
3.162	7.220	0.722	4.160	0.416

Very concentrated solutions are not very conductive. As they are diluted, the conductivity increases to reach a maximum concentrations of -log (c) = 5 to 6 M. Beyond the maximum 10^{-5} M, the conductivity decreases and tends practically towards zero.

An increase in the concentration can cause a decrease in solvation likely to lead to a lowering of the degree of dissociation, that is to say of the number of ions existing in the solution, as a result, without the volume of the solution varies, a decrease in the equivalent conductance (Λ). These findings lead to the conclusion that the concentration has a double influence on the conductivity: (i) the first is predictable, the conductivity linked to the presence of the electrolyte is normally proportional to the concentration. (ii) the second, equivalent conductivity acting in the opposite direction. At high concentrations, interactions between ions increase and decrease their mobility.

In this study the conductivity of the two systems increases with the increase in the concentration, in the absence of poly(ethylene oxide), our solution which is composed of LiI gives a conductivity equal to 4.16 S/m for an electrolyte concentration of 0.1M. On the other hand in the presence of poly (ethylene oxide), there is a strong increase in conductivity which is 7.22 S/m for the same concentration of 0.1 mol/l in electrolyte, the polymer solution to which we charge progressively electroactive species Li^+ and I^- greatly improve the conductivity of the medium, the ratio [σ (PEO) / LiI) / σ (LiI)] is 1.74. The ionic molar conductivity (Λ) behaves in the opposite direction to the electrical conductivity (σ). In aqueous solution, the molar conductivity of the isolated LiI electrolyte and in poly (ethylene oxide) (PEO) / LiI binary system decreases with increasing square root of the concentration (√C).

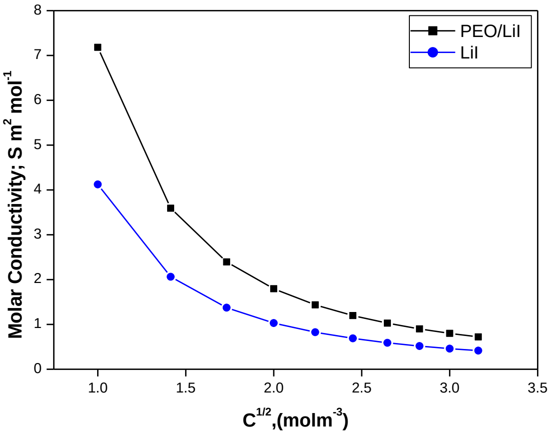

Figure 8. Change in molar electric conductivity as a function of the square root of the concentration for LiI and LiI + poly(ethylene oxide) solutions at 18 ° C.

In a slightly acidic solution pH = 6, poly (ethylene oxide) behaves like a cationic polyelectrolyte with a high charge density due to the protonation of the oxide groups. The conductivity depends on the number of charge carriers: it increases when the salt concentration increases. However, it also depends on the mobility of the ions: when the salt concentration increases, the viscosity of the electrolyte increases, the mobility decreases and therefore the conductivity decreases.

3.3.2 Effect of temperature

The conductivity of electrolytes solution is closely dependent on temperature and on the nature of the solvent. However, temperature is particularly important for the development of lithium-based battery systems, especially at low and high temperatures when considering the hot Sahara areas.

Fig.9 and Fig.10 shows the variations of electric conductivities as function of temperature (18 ° C, 25 ° C and 35 ° C) for lithium iodide LiI solution and lithium iodide (LiI) + poly(ethylene oxide) system. For an isolated solution of LiI (Fig.9), it is observed that the conductivity increases when the temperature increases. The variation is linear and the slope increases as the concentration of LiI increases. This observation differing to what we see in metallic conductors, the change in conductivity is not perfectly linear with temperature.

For this electrochemical system, it is preferable to determine the temperature drift coefficient (θ) over a small temperature ranges of 7 ° C, then over the entire range [18 °C to 32°C].The mean temperature drift coefficient θ over the entire interval is θ = 2.80 (% ° C^{-1}). It should be noted that for saline solutions, the values of the temperature drift coefficient θ vary from 2 to 3% for temperatures ranging from 18 to 32 ° C. Beyond a temperature of 32 ° C the variations in conductivity can be significantly different.

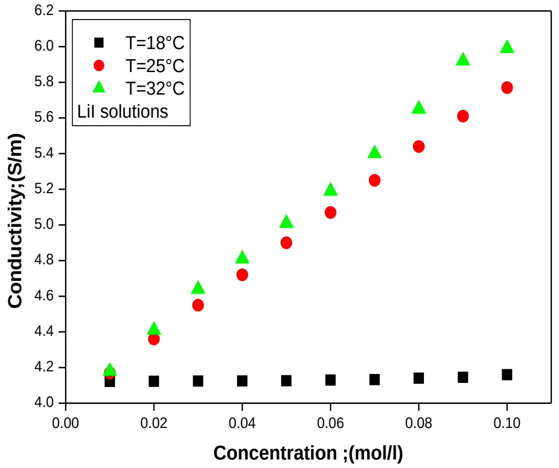

Figure 9. Evolution of the specific conductivity as function of concentration for different temperatures (18°C, 25°C and 32°C) for lithium iodide (LiI) solutions.

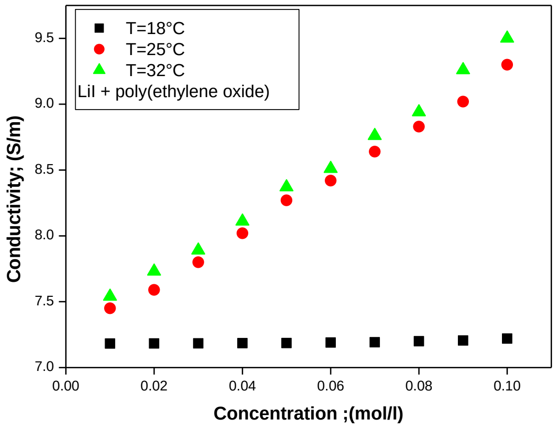

Figure 10. Evolution of the specific conductivity as function of concentration for different temperatures (18°C, 25°C and 32°C) for LiI/ poly(ethylene oxide) system.

Fig.10 clearly shows that the conductivity of the LiI/poly (ethylene oxide) solution system increases with the addition of the LiI salt as an electrolyte carrier. The polymer solution to which the électroactives species Li^+ and I^- are charged, greatly improves the conductivity of the medium by increasing the number of charge carriers. The electro-migration current is made up of the displacement of H^+ by about 73% and Li^+ cations towards the cathode by 11% and the displacement of I^- ions towards the anode by 16%. The increase of the specific conductivity (σ; S/m) of the solution as a function temperature is largely due to the increase in the mobilities of the Li^+ and I^- ions resulting from growing dissolution of the lithium salt which is an endothermic reaction, dissociation is shifted to the right with temperature and the number of charge carriers increases. In addition, the charge density of the positive cation Li^+ is very high than that of the anion I^-. The radii of cations are smaller than those of atoms. The positive charge density is very strong on Li^+, the cation is very solvated and its calculated hydrodynamic radius increases R_h = 2.40Å compared to its ionic radius R_{Na+} = 0.60Å, while the I^- ion is less solvated than the cation with a hydrodynamic radius R_h ≈ 1.67A °. Iodide ion I^- conducts more and diffuses faster.

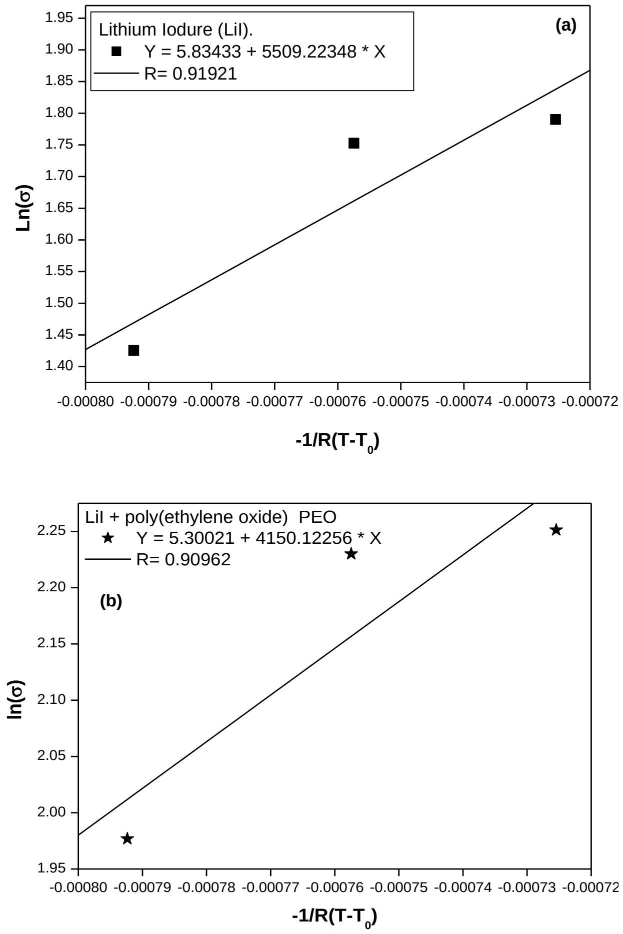

Figure 11. Linear correlation curves according to the Vogel-Tamman-Fulcher model for (a) LiI solutions and (b) LiI + poly (ethylene oxide) system using 0.1M LiI and T=18°C -32°C.

3.3.3. Activation energies

Applying the empirical Vogel-Tamman-Fulcher (VTF) equation allows us to find the activation energy (Ea) as well as the factor (A) of the electrochemical system [20, 21].

$$Ln(\sigma) = Ln(A) - \frac{E_a}{R(T-T_0)} \qquad (4)$$

σ is the electrical conductivity (S/m);
A is the pre-exponential factor;
Ea is the activation energy (J/mol);
R is the ideal gas constant (J/K);
T is the temperature (K) and T_0 is the temperature of the glass transition (for water T_0 = 140 ° K).

The linear regression results according to the Vogel-Tamman-Fulcher model for the LiI/poly (ethylene oxide) PEO and the isolated LiI systems considering differents temperatures are showed in Fig.11a and Fig.11b, respectively. From the results of Fig.11a and Fig.11b, we see that the slopes of the lines influence the values of activation energies and the pre-exponential factors. The use of poly (ethylene oxide)/electrolyte solution achieves relatively high conductivities with increasing temperatures.

According to the Vogel-Tamman-Fulcher equation (VTF), three parameters have an important influence on the electric conductivity: (i) the activation energy (Ea);(ii) the parameter A which has the dimension of the conductivity;(iii) the temperature at zero mobility (T_0). Thus, to have considerable electric conductivities, the electrochemical system used must imperatively ensure the lowest possible activation energy with a high electrical conductivity correlated with a high ionic mobility resulting from a low viscous electrolyte system allowing good salts dissociation.

The main parameters of the VTF equation determined for the two electrochemical systems are given below (table2):

Table 2: Main parameters of the VTF

Systems	LiI + PEO	Lithium Iodure (LiI)
Ea (KJ/mol)	4.15	5.51
A(S/m)	200	342

4. Conclusion

In the present work, the electric conductivity of systems based on poly (ethylene oxide) PEO and without PEO of seven binary solutions (KCl, NaCl, NaOH, LiCl LiBr, LiI and $NaNO_3$-H_2O) and of three ternary Halogenated lithium solutions (LiCl-H_2O, LiBr-H_2O and LiI-PEO-H_2O) were measured as a function of changes in concentration and temperature. The following conclusions are drawn: (i) the addition of an electrolyte solutions to the poly(ethylene oxide) PEO polymer causes an increase in the specific conductivity (σ; S m) by a factor of two. For the same solvent and under the same concentration and temperature conditions, the electric conductivity is reduced in the order: LiI-water> LiBr-water> LiCl-water;(ii) In binary and ternary systems, halogenated lithium salts (LiCl, LiBr and LiI) showed the highest values of specific conductivity. A maximum of the conductivity value is observed especially for poly (ethylene oxide)/ lithium iodide (LiI) / water and lithium iodide (LiI) / water systems. At 18°C the maximum electric conductivities were 7.01 S/m and 3.95 S/m, respectively; (iii) The electrical conductivity of polymer solutions solutions are influenced by the characteristics of the electrolytes solutions, it increases with its ionic radius (Å) and its ionic mobility (u; ($m^2\ s^{-1}\ V^{-1}$));(iv) In the range of the studied concentrations [0.01M to 01M], the electric conductivity of polymer solutions increases as the concentrations of the electrolytes increases; however, the rate of increase is relatively small at low concentrations. The electrical conductivity increases linearly and beyond 0.5M the conductivity doubles in value;(v) The conductivity of the polymer ternary solution system (LiI / poly (ethylene oxide) / water) and the electrolyte in the binary system (LiI / water) increases linearly with increasing temperature;(vi) The Vogel-Tamman-Fulcher (VTF) equation is used to correlate the conductivity as a function of the temperature variation for the ternary systems (poly (ethylene oxide) / Lithium iodide (LiI) / water) and binary (Lithium iodide (LiI) / water). The deduced activation energies were Ea = 4.15 and Ea =5.51 KJ / mol. The ternary solution of LiI-poly (ethylene oxide) PEO -H_2O has a real potential as working solutions for Lithium-Ion batteries due to their electrochemical and thermodynamic properties.

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