D.C. Electrical Properties of $MgCl_2$–Filled PEO Films

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Abstract

The D.C. electrical properties of poly (ethylene oxide)/$MgCl_2$ composites were investigated as a function of different $MgCl_2$ filler concentrations (0, 5, 10, 15 and 20 wt.%) and different temperatures in the range (276–333)^{o}K at three different polarizing fields. Resistivity: ρ and dc Conductivity: σ_{dc} were measured, and the activation energy: E_a of the thermal rate-process of the electrical conduction was investigated. It was found that the current-voltage measurement results exhibited Ohmic resistance behavior, the composites exhibit negative temperature reliance of resistivity and enhancement in the D.C. electrical conductivity with both temperature and $MgCl_2$ concentration. The determined activation energy was found to decrease with both applied polarizing fields and $MgCl_2$ content. The observed overall mechanism of electrical conduction was discussed on the basis of mobility of PEO chains and to the transfer of electrons through the salt aggregations distributed in the polymer matrix, and it was observed that all of the composites were found to be similar to the semiconducting behavior.

Keywords: DC Conductivity; PEO; $MgCl_2$; Activation energy.

Introduction

Materials can be classified according to their electrical conduction into: conductor, semiconductor and Insulator; one of the most important advanced subject, namely conductive polymer composites, was given a great interest in many industrial applications and covering large area in different material uses owing to their desirable properties which made them favorable compared to other commercial materials [1]. Conducive polymer composites possess a wide range of electrical conductivity that covers several orders of magnitude extending from insulators up to semiconductors and sometimes to good conductors [1, 2]. Semiconducting polymeric mixtures are becoming common in many applications such as heating, prevention of static electricity accumulation, for example, preventing dust attraction on Computer cabinets and electrodes [2, 3]. Polymers in semiconducting/conducting region are used for electromagnetic interference shielding of electronic devices and preventing of static electricity hazards in the handling of electronic chips and explosives [3]. Conductive polymers are also used as sensors, heating elements and particularly selflimiting electrical heaters, switching devices and optoelectronic applications [4].

Electrets formed by thermal methods are referred to as thermoelectrets. It is well known that in general, thermally stimulated discharge conductivity from thermoelectrets is considered to be induced by the thermal release of dipoles, ions and trapped electrons [1, 5], and the conductivity of the composite is determined by the combined effect of three main mechanisms: (a) quantum tunneling or hopping of electrons through a thin insulating film that can form on the surface of the conducting filler; (b) constriction of electron flow due to particle–particle contact resistance; and (c) intrinsic conduction of the filler [6].

There are two approaches to make electrically conducting polymer composites: (i) use of conductive (salt or carbon black fibers or particulate fillers) in appropriate lay up, doses and dispersions in an insulating polymer matrix to produce semiconducting or conducting reinforced or filled composites and (ii) use of an inherently conducting polymer intimately blended with or dispersed in a second polymer matrix (i.e. insulating in character) during or subsequent to synthesis [4, 7].

The present work belongs to category (i) and accounts for the experimental study relating to the behavior of d.c. electrical conductivity in insulating polymer like PEO rendered semiconductive with filling of less amount of $MgCl_2$ salt. Highly dispersed activated salt, giving sufficient conductivity at comparatively low concentrations، is used. The D.C. electrical conductivity and the temperature effect on resistance were studied as a function of $MgCl_2$ concentration and temperature using the current/voltage characteristics.

Many kinds of conductive fillers have been used with insulating polymers such as salt, carbon fibers, and metallic powders. Inorganic salt is a material that has found widespread use in a number of applications; the purpose of adding $MgCl_2$ filler is to improve the physical properties of polymers, where $MgCl_2$ with high structure has a high ability to form a continuous network in the polymer matrix and hence leads to higher conductivity [8, 9]. Many studies have been reported on electrical and thermal properties of PEO-salt composites which might be useful in some advanced and industrial applications [10, 11, and 12]. On the other hand, investigation of the D.C. electrical properties of these composite systems is still limited. At the same time, to our knowledge, no research reported the effect of $MgCl_2$ salt on the D.C. electrical properties of PEO polymer [8,13], AYESH et al. [14]studied the electrical properties of PC/$MnCl_2$ composite and they found that the volume resistivity decreases with the increase of $MnCl_2$ concentration while the D.C. conductivity is in the range of 10^{-5} $(\text{ohm.m})^{-1}$.

These filled polymers have a number of advantages in terms of (i)absorbing the specific radiation, (ii)improving thermal stability, (iii)enhancing thermal and electrical conductivities, and (iv)reducing the cost and easy processability to achieve conductivity [15]. The study of resistivity and thermal noise of amorphous polymers (Poly ethylene oxide, Poly ethylene glycol) contains a small percentage of NaI which was reported by Dillip et al [16]. Time dependent resistivity was recorded in glass transition (Tg) and melting temperature (Tm) regions.

The thermally stimulated discharge current technique which is used to study the D.C. electrical conductivity properties, proved similarity between dipolar and space charge relaxations and it is a basic tool to identify and evaluate the dipole reorientation process, trapping and recombination levels in electrets [1, 17].

Experimental

Films preparation

The resin used in this work is poly (ethylene oxide) resin (MW 4,000,000) was obtained from CNR (Nopoli-Italy). Ordinarily, the salt $MgCl_2$ was ground into fine powder by agate mortar and sieved by a U.S. standard sieve of size (63 µm). Thin films of PEO/$MgCl_2$ were prepared by dissolving PEO resin in methanol at 303 ^{o}K; the $MgCl_2$ salt was also dissolved in methanol at the same temperature. Both solutions were mixed together for one hour by using a magnetic stirrer until a homogenous solution is obtained. Then the mixture was cast into a stainless-steel ring resting on Teflon substrate and waiting for a few days until the solvents have evaporated. Samples of different concentrations were dried in vacuum oven at 313 ^{o}K for two days. The drying process was repeated until prepared membranes have fixed weight to ensure the removal of solvent traces and to ensure uniform thickness. Then after completing evaporation, the film was detached from ring surface. In this way, the films were prepared by solution evaporation technique (casting method). The same procedure was used in the researches [8, 11, 13, and 14].

(PEO + $MgCl_2$) , were prepared in the laboratory by weight percent method with an accuracy of 0.0001 g. Electronic single pan balance, Adiardutt-180. The prepared films contain 0, 5, 10, 15 and 20 wt% $MgCl_2$. The film thickness (~0.15 mm) was measured using a ballended micrometer.

Electrode coating

For good ohmic contact, both the surfaces of film were coated by quick drying and highly conductive Silver paint (Ernest F. Fullam Inc., Latham, NY) was applied to contact points in order to reduce the resistance between the electrodes and the sample surfaces.

Electrets preparation

The experiments were done in winter, cause decrease in temperatures. The coated film was sandwiched between the two copper electrodes shielded cell, and the applied pressure to hold the test specimen was exerted by top and bottom electrodes. The sample holder forming metal-polymer-metal system was placed in a furnace and heated up to the 340°K. The sample was allowed to remain at that temperature for about 30 min. Then electric field of desired strength was applied for 1 hour at this temperature with the applied electric field. The sample was allowed to cool down to 276^{o}K in the presence of applied field. Total time of polarization was adjusted to be 2 hour in each case. On attaining 276^{o}K, the samples were kept shorted for 20 min to eliminate the stray charges. The electrets were prepared at different D.C. polarizing fields: E = 300 V/cm, 700 V/cm, 1100 V/cm, respectively.

The D.C. Electrical Conductivity Measurement

After electrets formation, the test sample holder assembly was placed in a controlled temperature furnace. The D.C. electrical conductivity was measured by determining the resistance of a sample within temperature range 276-333 °K at the rate of 2°/min. The temperature was recorded by a digital thermometer having an accuracy of ±1°C. The electrical input was provided by means of a stabilized D.C. power source, and the current passing through the specimen was measured by a digital multimeter (Model 3458A, Hewlett-Packard, Houston, TX). A digital multimeter (systronics, 435) having an accuracy of ± 1 mV was used for the measurement of voltage drop across high resistance. The current and temperature were monitored continuously during measurements. The method used for conductivity measurement was the same as that reported earlier with Shingo *et al* [18] and the sketch for the setup and the electrical circuit diagram used in the D.C. measurements was recently reported [19]. Note that the volume resistivity ρ□$_v$□□□ was determined from the well-known relation equation [20]:

$$\rho_v = \frac{A}{\delta} R_v \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where (R_v) is the volume resistance between the guarded and the bottom electrodes, which was measured directly by obtaining the current-voltage characteristics of the cell; (A) is the disc area of the electrodes of radius 0.5 cm; and (δ) is the average thickness of the sample (~0.15mm).

Results and Discussion

The results of the present study are in the form of thermograms {Figures 1–2–3} which are curves between log of thermally stimulated discharge conductivity, log (σ_{dc}) and temperature (10^3/T) of the sample films at different polarizing fields. Where, a significant curvature is quite pronounced, this curvature has been frequently observed in many amorphous polymer composites systems [21].

The thermograms of Figure 1 are of pure PEO film. For all polarizing fields, the D.C. conductivity is in insulating order. Figures 2 and 3 show that the PEO was rendered semiconductive by the addition of 0.05 and 0.20 weight percent of $MgCl_2$. The slight decrease is observed at low temperature and continuous increase is observed up to 330°K in all the samples. A nonlinear field dependence is clearly evident in pure and filled PEO samples.

The constituent of pure film, viz. PEO, is largely an amorphous polymer. It is characterized by three relaxations: β relaxations occurring at low temperature, α relaxation around the glass rubber transition temperature (T_g) and α1 relaxation occurring at a temperature well above (T_g) [22,23] . The absence of peaks in the present thermograms might be due to low applied electric field [17].

In polymeric materials, various types of molecular relaxations are possible. The only motions possible at a low temperature are local motions of molecular groups, e.g. rotation of side groups or internal motion within the side groups. Hence, at low temperature there may be slight decrease and then rise in conductivity of thermoelectric of pure PEO films. This is due to mobility of main chain segment increase with the increase in temperature [6, 8].

Figures 2 and 3 represent thermograms (log σ_{dc} vs. $10^3/T$ plot) of 0.05 and 0.20 weight percent $MgCl_2$ salt added PEO thin film thermoelectrets for different polarizing fields (300V/cm, 700V/cm, 1100V/cm). In both of the cases, the conductivity increases with increasing temperature i.e. semiconducting nature of thermoelectric. Our experimental study revealed that electrical conductivity increases with the increase in temperature approximately by the following equation [24]:

$\sigma = \sigma_0 \exp(-E_a/RT)$ ……………… (2),

Where σ is conductivity, σ_0 the pre-exponential factor, E_a the activation energy of conduction and R the universal gas constant. The activation energy, Ea, was calculated from the graph of log σ_{dc} vs. $10^3/T$ plot. Tables 1–3 show the variation of activation energy values within low, intermediate and high temperature regions.

Addition of $MgCl_2$ increases the conductivity of polymer considerably {Figures 2 and 3}. As the percentage of loading is only 0.05 weight percent in second case, smooth increase was observed. Initially increase in conductivity at low temperature may be due to the injection of charge carriers directly from the electrodes [25, 26]. The filler is very sensitive to temperature. There are two phases i.e. polymer phase and filler phase. They formed heterocharges and discharge by dipole disorientation is thermally activated and so can be speeded up by heating. The increase in conductivity at higher temperature may be due to softening, the injected charge carrier can move more easily into the volume of the sample giving rise to a large current and increase in conductivity at higher temperature [6, 25].

In the third case, as shown in thermogram 3, the loading of $MgCl_2$ Salt is slightly increased from 0.05–0.20 weight percent. There is a slight change in conductivity,(σ_{dc}) for these samples especially at higher temperature above T_g. As the temperature increases, the chain of the PEO becomes more and more flexible [1]. This conduction is mainly due to a direct contact with Salt particle as explained by Tawansi et al and Saq'an et al [8, 11].

It is very clear at the first Figures {1–2–3}, the D.C. conductivity increases with the increase of both electrical field and temperature. The increase in conductivity with temperature at a sensitively low field is due to the increase in the magnitude of the main free path of phonon [8, 25]. At high temperature enhancement in conductivity is mainly attributed to the increase in ionic mobility of $MgCl_2$ Salt particles [24]. Our results are in a good agreement with Tawansi *et al* [8], Jovic′ *et al* [6] and Ramadin et al [27].

The activation energy decreases significantly on the increase of the D.C. electrical field at room temperature as shown in Figure 4 for different $MgCl_2$ Concentrations, to emphasize the possible effects of D.C. electrical field value. The field dependence of D.C. conductivity, σ_{dc} of the composites 5%wt $MgCl_2$ and Pure PEO is similar. In both cases, the conductivity increases sharply with increasing electric field. However, as mentioned above, both curves are much more sensitive to electrical filed than that 20wt.% $MgCl_2$ sample is almost unaffected by increasing the electrical field. This can be probably ascribed to the enhanced electron scattering at filler contacts due to an increase in their kinetic energy as the electric field increases [6, 28].

For solid-state D.C. current-Voltage (I–V) measurements of PEO/$MgCl_2$ composite, Figure 5 represent the D.C. voltage/current characteristics at different temperatures for the 5 wt.% $MgCl_2$ composite, and exhibits a linear relation indicating ohmic behavior of the materials, which is consistent with electronic conduction [4]. The D.C. conductivity was evaluated to be 1.7×10^{-5} S/m. The enhancement in the conductivity is by approximately two orders of magnitude compared to the pure PEO and there is a small change in the conductivity with temperature. It was observed earlier that the PEO-$MgCl_2$ composite showed very low resistance and the enhancement were attributed to inter particle tunneling [20].

Figure 6 shows the variation of the Resistivity (ρ_v) with $MgCl_2$ salt concentrations. At lower concentrations -below 10 wt%- it was observed that the electrical resistivity decreases sharply; at higher salt concentrations, the resistivity decreases slightly where the composite becomes a good conductive substance at salt contents higher than 20wt% . This transformation from an insulating material to conductive one is due to the semiconductive $MgCl_2$ salt and the generated electrical contacts resulting from the salt networks [18].

Figure 7 represents the D.C. electrical resistivity as a function of temperature for composites with different $MgCl_2$ salt concentrations. It was found that, at low salt concentrations (0, 5 and 10 wt %), the resistivity of samples is high and decreases sharply with increasing temperature, which indicates that the D.C. electrical resistivity varies similar to a semiconducing behavior. The observed behavior in the D.C. electrical resistivity with $MgCl_2$ salt concentration is attributed to the percolation theory, where polymer/conducting-filler composites exhibit a sudden transition from insulator to conductor at a certain filler concentration less than 10 wt% $MgCl_2$. This sudden transition in the composite resistivity is a characteristic of percolation threshold [21, 29]. At high salt concentrations over than 15wt.% $MgCl_2$, the resistivity decreases slightly and slowly over the testing temperature range, This result indicates a metallic-type conduction mechanism due to conductive networks of Salt, where the thermal expansion effects cause a reduction in electrical contacts between the salt particle networks and hence result in a slowly decreasing resistivity of the composite with temperature [30], or this slight decrease in resistivity may be due to the space charge effect caused by the addition of Salt [25]. All tested composites of different $MgCl_2$ concentration showed negative temperature coefficient of resistivity.

Figure 8 shows the variation of the DC-conductivity (σ_{dc}) with temperatures for different salt concentration in the PEO/$MgCl_2$ composite. It was observed that the D.C. conductivity increases with the increase of both temperature and $MgCl_2$ concentration. The increment in temperature provides an increase in free volume and segmental mobility [15, 31]. These two entities then permits free charges to hop from one site to another thus increase conductivity. The conductivity increases so as temperature indicates more ions gained kinetic energy via thermally activated hopping of charge carriers between trapped sites, which is temperature dependence [32]. The observed increase of D.C. conductivity between 300 to 340 ^{o}K can be attributed to large heat energy absorbed by the samples and thus induce mobility of electrons [32]. It is suggested that in this region, the band gap between valence band and conduction band is reduced significantly and provide easiness for electrons to hopping from valence band to conduction band [1, 33] and hence gives higher D.C. conductivity values as compared to other temperatures., i.e. The conductivity of the polymer/Salt composites strongly depends on the particles' interfacial resistance, which has two major contributions: ''contact resistance'' and a so-called ''tunneling resistance''. The former dominates in highly filled composites, when physical contact between the particles is present, while the latter is related to the presence of an insulating polymer film on the particle surfaces. Tunneling is a transport process that depends on thermal fluctuations [34], and a temperature increase will reduce ‘‘tunneling resistance”, i.e. give rise to conductivity [6, 35], it is probably the result of the separation of the particles due to the softening of the PEO matrix. It appears, however, that the ‘‘tunneling conduction” can compensate for the lack of particle contacts because at higher temperatures the energy barrier tends to be lower [34].

Figure 9 represents Logarithm of resistivity {Log□□ (ρ_v)} against reciprocal absolute temperature, or what is called the Arrhenius exponential law equation (15, 36). The activation energy Ea is calculated from the slope of the best fit of the experimental Arrhenius curves as approximate straight lines by using the equation:

ρ_v = □□/σ.........(3)

Where ρ_v is the measured resistivity and σ is conductivity which is shown in equation [2]. The conduction activation energy of the conductivity influenced by temperature is the minimum energy required to overcome potential barrier in the composite system.

The activation energies for the composites calculated from Figure 9 are shown in Table 4 with average error (±0.027), the dependence of the apparent activation energy of the electrical thermal activated rate-process on the $MgCl_2$ salt concentration is represented in Figure 10. The figure shows that the activation energy decreases rapidly with the increase of the $MgCl_2$ salt concentration of composites. This behavior of the activation energy with temperature is similar to most conductive polymer composites with Salt content [8, 11, 12, 18, 19, 37, 38], a case which reflects that a new conductive polymer composite is constructed from the insulating polymer PEO.

Finally, it is worth mentioning that our calculated value of the activation energy is about 64 kcal.mol^{-1} for pure PEO, and it is very close to the value of 65 kcal.mol^{-1} as recently reported in [39].

Conclusion

A study of the effects of the filler salt concentration and temperature on the volume D.C. conductivity of PEO/$MgCl_2$ composite is presented in this article. The volume resistivity behavior and the concentration of salt properties of this composite are investigated at temperatures ranging from (276 to 333) ^{o}K. From the obtained results, the following conclusions are drowning:

1. Both the temperature and addition of $MgCl_2$ Salt concentration influence the D.C-conductivity of PEO; this filled PEO polymer could be good for low cost semiconducting composites.
2. The increase in temperature (above T_g) would lead to the increase in D.C. conductivity, and the increase in the electrical field would lead to a decrease in the activation energy.
3. The resistance of the PEO/$MgCl_2$ composites was found to be Ohmic, and the temperature coefficient of resistance is negative.
4. The overall dependence of the D.C. electrical conductivity of the PEO-$MgCl_2$ composites on temperature was found to be similar to the semiconducting behavior.
5. The temperature dependence of D.C. conductivity for the given composites is strongly influenced by the conductivity behavior of the $MgCl_2$ concentrations.
6. The overall conduction mechanism is related to electrons transfer through the $MgCl_2$ aggregations distributed in the polymeric matrix.
7. The increase in both of temperature and $MgCl_2$ concentration would lead to a clear decrease in the activation energies of the composites.
8. The thermoelectrically conduction behavior of the composites is interpreted in the form of variable range hopping mechanism and on the basis of mobility of PEO chains and to the transfer of electrons through the salt aggregations distributed in the polymer matrix.

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Table (1): Variation of activation energy, E_a, in the three temperature regions for pure PEO thin film.

Polarizing field (V/cm)	E_a (kcal. mol^{-1})	E_a (kcal. mol^{-1})	E_a (kcal. mol^{-1})
300	59.3425	63.9917	68.7786
700	45.7856	49.9293	53.9987
1100	39.5662	43.2245	40.6322

Table (2): Variation of activation energy, E_a, in the three temperature regions for 5wt.%$MgCl_2$ doped PEO

Polarizing field (V/cm)	E_a (kcal. mol^{-1})	E_a (kcal. mol^{-1})	E_a (kcal. mol^{-1})
300	35.5623	43.9928	49.1129
700	26.7783	33.1255	39.3335
1100	21.2257	24.6503	28.9664

Table (3): Variation of activation energy, E_a, in the three temperature regions for 20wt %$MgCl_2$ doped PEO

Polarizing field (V/cm)	E_a (kcal. mol^{-1})	E_a (kcal. mol^{-1})	E_a (kcal. mol^{-1})
300	5.9753	6.2403	4.5465
700	1.1411	2.2595	2.0042
1100	0.2566	1.4177	2.4571

Table (4): The Variation of the activation energy with $MgCl_2$ concentration.

Sample	Activation Energy, E_a (kcal. mol^{-1})
PEO (Pure)	63.99178
PEO + 5 wt.% $MgCl_2$	43.99282
PEO + 10 wt.% $MgCl_2$	17.46373
PEO + 15 wt.% $MgCl_2$	8.674247
PEO + 20 wt.% $MgCl_2$	6.24029

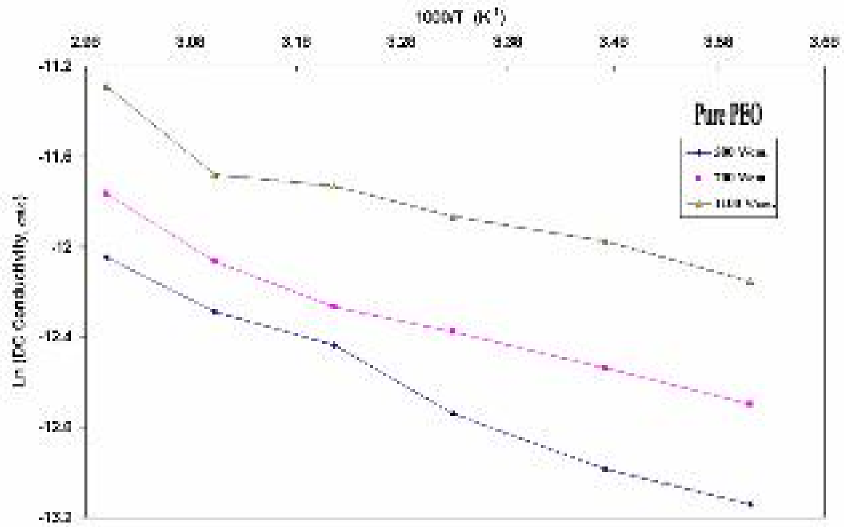

Fig. (1): Natural Logarithm of DC-Conductivity vs. 1000/T for Pure PEO. (at different d.c. polarizing fields)

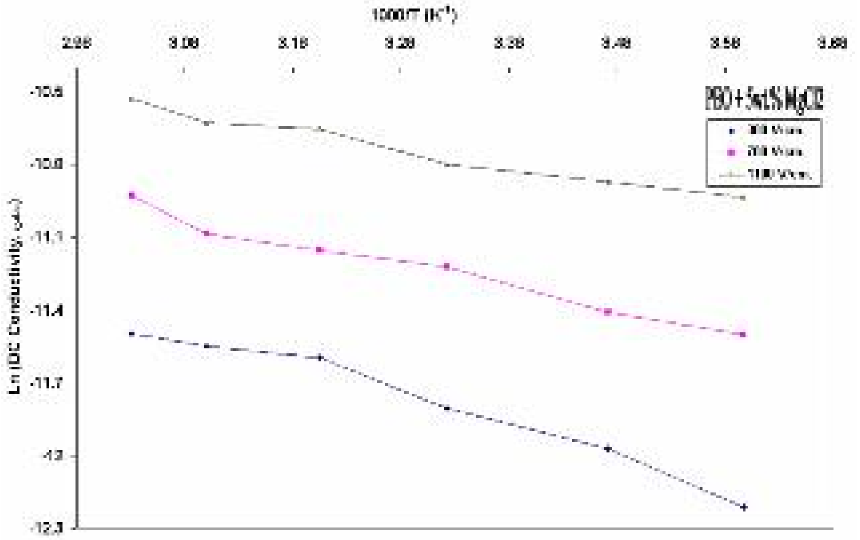

Fig. (2): Natural Logarithm of DC-Conductivity vs. 1000/T for 5wt.% $MgCl_2$ (at different d.c. polarizing fields)

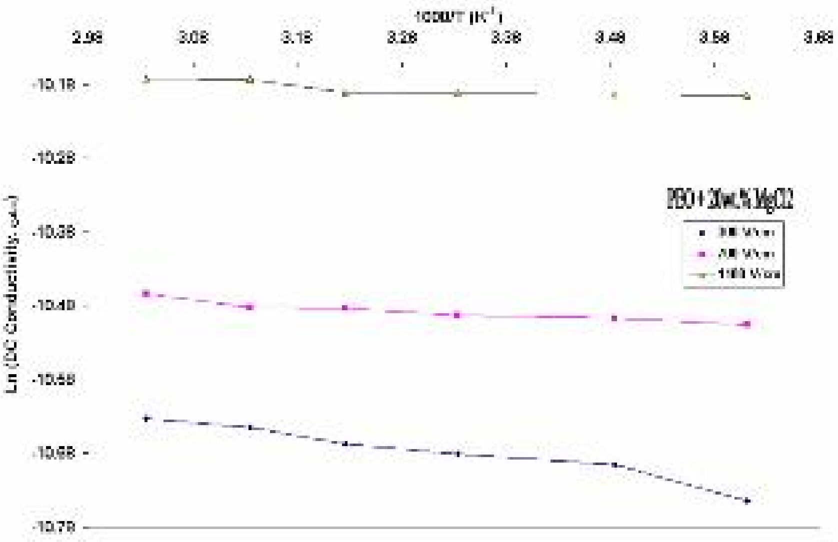

Fig. (3): Natural Logarithm DC-Conductivity vs. 1000/T for 20wt.% MgCl$_2$ (at different d.c. polarizing fields)

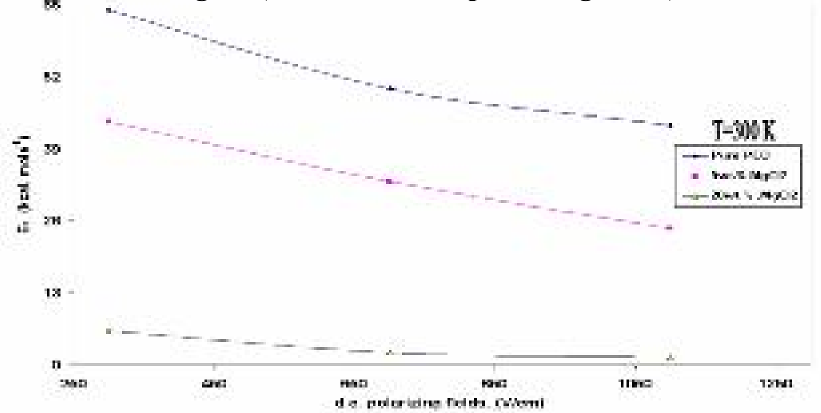

Fig. (4): Variation of the activation energy with DC-field (for different MgCl$_2$

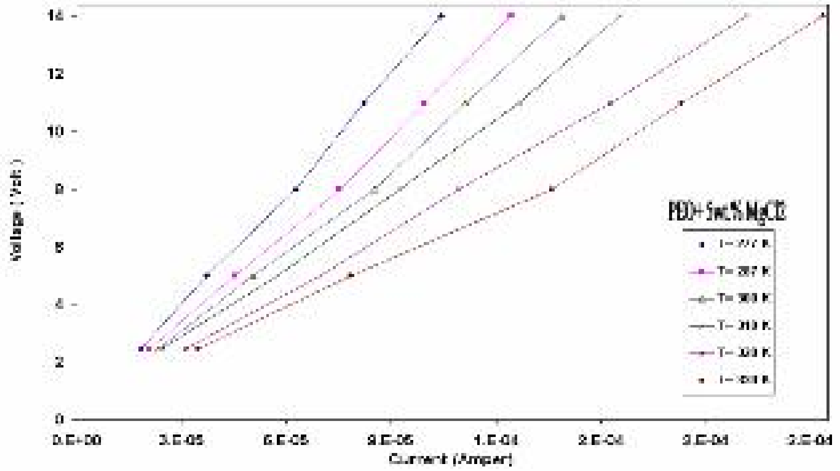

Fig. (5): V–I characteristics for the 5wt.% MgCl$_2$

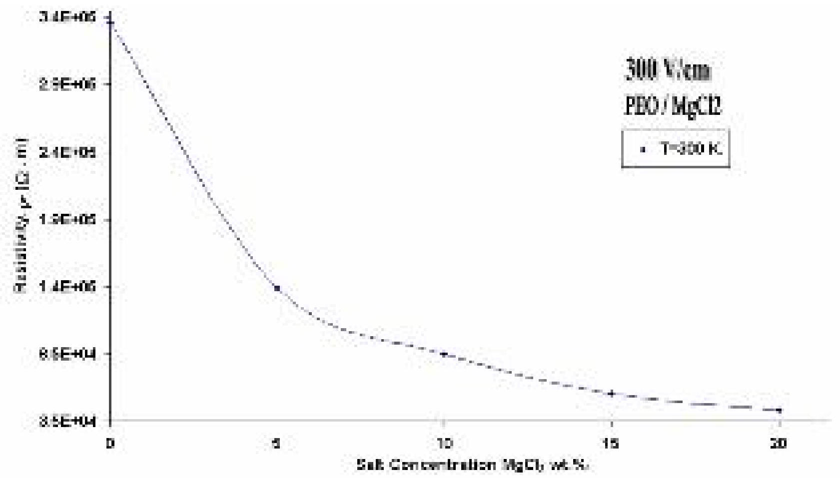

Fig. (6): Volume Electrice Resistivity as a function of MgCl$_2$

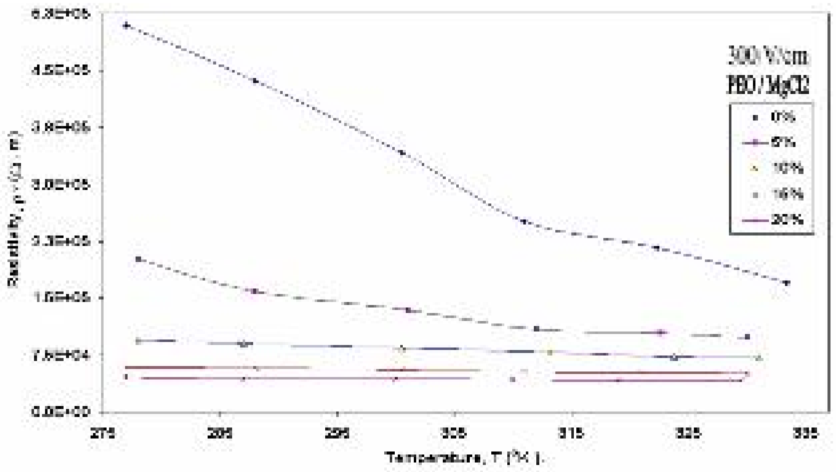

Fig. (7): The Volume Electrical Resistivity as a function of temperature.

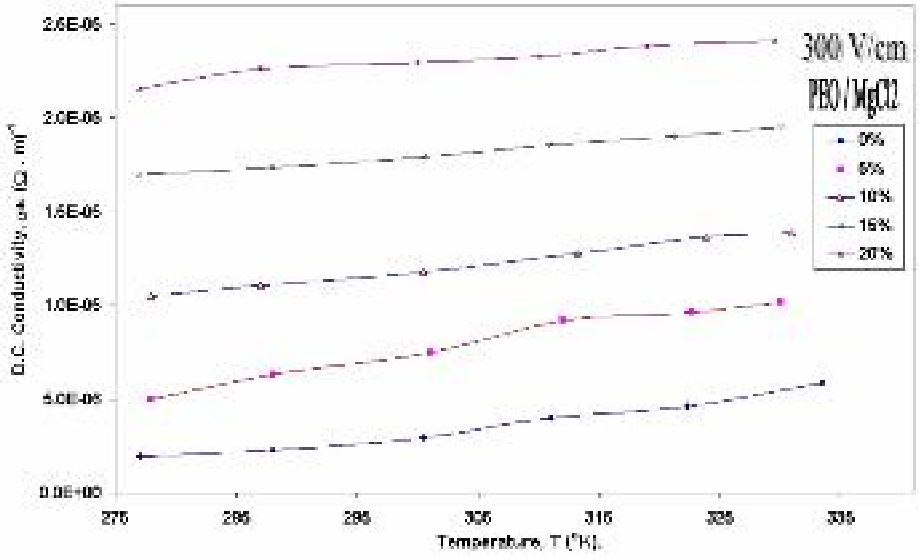

Fig. (8): The DC-Conductivity as a function of Temperature.

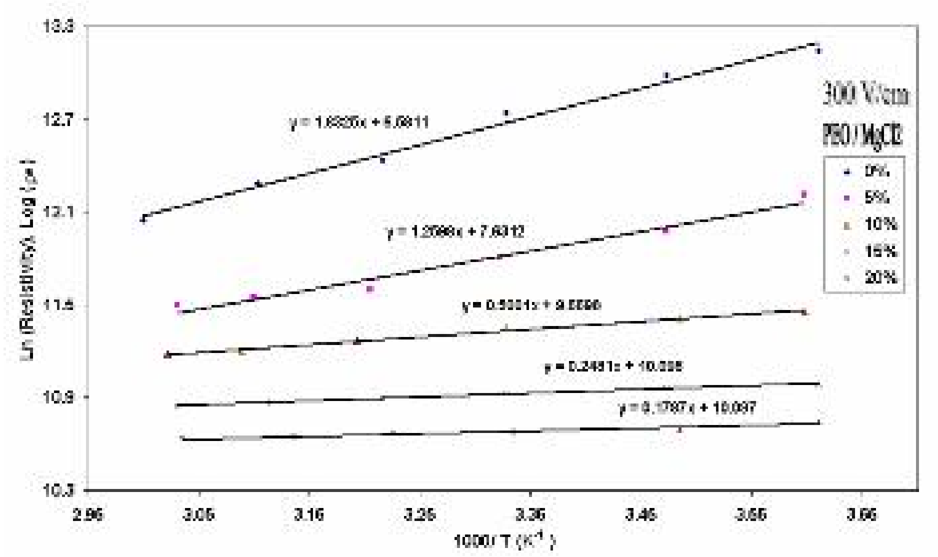

Fig. (9): The Volume Electrical Resistivity Natural Logarithm versus the Reciprocal Temperature.

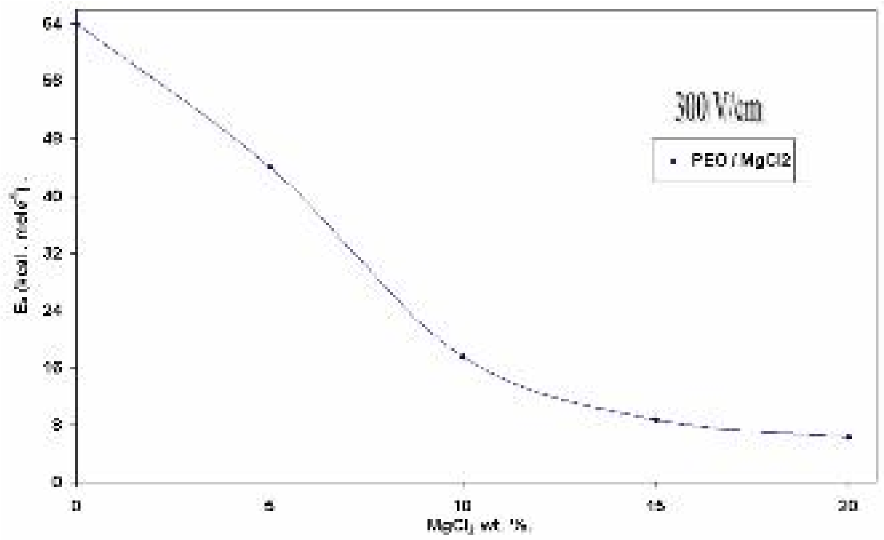

Fig. (10): Variation of the activation energy with the $MgCl_2$ Concentration.

الخواص الكهربائية المستمرة للأغشية المتكونة من بولي أوكسيد الإثيلين مع الملح كلوريد المغنسيوم

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الخلاصة

تم في هذا البحث دراسة الخصائص الكهربائية المستمرة لمتراكب من (بولي أوكسيد الإثيلين) قالباً وملح (كلوريد المغنسيوم) حشوة ذا تراكيز مختلفة داخل القالب بالمدى (0% إلى 20%). تعاملت هذه الدراسة مع التوصيلية الكهربائية المستمرة للمتراكب دالة لتراكيز الملح و درجة الحرارة بالمدى (276 درجة كلفن إلى 333 درجة كلفن) وضمن ثلاث مديات من مجال الأستقطاب المسلط على العينة.

لقد حُسبت المقاومة الكهربائية النوعية و قيست التوصيلية الكهربائية المستمرة وكذلك طاقة التنشيط الحرارية. لقد وُجد من النتائج أن نتاتج قياسات تيار – فولت تبدي سلوك مقاومة أومية، وكذلك المتراكب يبدي مقاومة كهربائية عكسية مع زيادة درجة الحرارة، وأن زيادة كل من درجة الحرارة وتركيز الملح تزيد التوصيلية الكهربائية المستمرة. وتنخفض طاقة التنشيط الفعالة مع زيادة كل من مجال الأستقطاب الكهربائي المسلط و تركيز الملح في المتراكب . ويعزى تفسير معظم هذه الظواهر إلى قواعد حركة سلاسل المبلمر و إلى أنتقال الألكترونات بين تجمعات كتل الملح المنتشرة في قالب العينة، و قد أظهرت جميع العينات سلوك مشابه لسلوك المواد الشبه موصلة الكهربائية.

الكلمات المفتاحية: التوصيلية الكهربائية المستمرة، المبلمر بولي أوكسيد الإثيلين، الملح كلوريد المغنسيوم، طاقة التنشيط الفعالة.