Electrical Properties of Poly (Ethylene Oxide) polymer Doped by $MnCl_2$ [*]

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Abstract

The electrical properties of Poly (ethylene oxide)-$MnCl_2$ Composites were studied by using the impedance technique. The study was carried out as a function of frequency in the range from 10 Hz to 13 MHz and $MnCl_2$ salt concentration ranged from 0% to 20% by weight. It was found that the dielectric constants and the dielectric loss of the prepared films increase with the increase of the $MnCl_2$ concentration; The A.C. conductivity increases with the increase of the applied frequency, and the $MnCl_2$ content in the composite membrane. Relaxation processes were observed to take place for composites which have a high salt concentration. The observed relaxation and polarization effects of the composite are mainly attributed to the dielectric behaviour of the $MnCl_2$ filler and polarity of the polymer PEO. However, the results were explained on the basis of the interfacial (space charge) polarization dipolar polarization and the decrease of the hundrance of the polymer matrix with the ionic mobility and impurities in the composite.

Keywords: Electrical properties; PEO matrix; $MnCl_2$ filler; Composite; Impedance; Field frequency; Dielectric constant; AC-Conductivity; Polarization.

Introduction

Polymeric materials were given a greet interest in many industrial applications owing to their desirable characteristics and properties which made them favorable compared to other commercial materials .The vast majority of polymers used today as plastics, rubbers, adhesives and paints which are synthetic petrochemicals [1]. The unbeatable combination of characteristics such as the ease of fabrication, low cost, light weight, ease of chemical modification and excellent insulation or good conduction properties have made the polymer one of the most desirable materials for application [2].

Many studies showed that physical properties of polymers clearly depended on many factors concerning their preparation methods and chemical structure [3]. Understanding these dependencies and their effect on conduction mechanism will help to a large degree the ability for controlling the electrical conductivity, which is in turn trial the proper application. Poly (ethylene oxide) or (PEO) is a crystalline, homopolymer with general formula $(-H_2C-O-CH_2-)_n$. PEO polymer is a thermoplastic water-soluble and in several organic solvents. The molecular conformation of it is determined by the use of X-ray diffraction techniques. The diffraction pattern of highly printed (PEO) film was interpreted in term of a monoclinic unit cell have dimensions, a = 7.96 A^o , b= 13.11 A^o and c = 19.39 A^o along the direction of applied stretch, and an angle of $124^o.48`$ representing the inclination between the a&c axis as shown in picture (1).

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The thermal stabilities of crystalline PEO-$MnCl_2$ system depend on the salt molar ratio, the PEO molecular weight, the choice of the solvent and the concentration, and the thermal history. The melting temperatures also, depend on the nature of the complexion salts. [4]

The PEO polymer has a wide range of application including the use as pharmaceutical recipients, food additives and plasticizers [5]. However, much progress was made in the electrical conduction in polyethylene (PEO) since the work of Wright [6]. Previous studies were centered on the enhancement of its ionic conductivity with the aim of developing the material to have the promising electrical application [6,7].

Considerable efforts focused on an applied research in the field of polymer composites to turn these materials into useful products for electronic industry. This is mainly because they possess interesting properties which can be utilized to develop a lot of related potentials. Recently, many reports have appeared in literature dealing with the effects of the filler concentration, frequency of the applied field and temperature on the physical properties of the conductive polymer composite such as impedance, dielectric behaviour and electrical conduction [8,9,10].Jamali and Zihlif [11] studied the electrical properties of PEO treated by salt complexes of Dead Sea Water as KCl, NaCl and others.

They found that salt complex enhances the electrical conductivity through the ion conduction process. Ramadin and Zihlif et al [12] studied the optoelectrical properties of PEO containing 10, 20 and 30% by weight Alum and they found that the optical energy gap decreases with the increase of the Alum content. Eid [13] studies the effect of temperature, frequency and PEO concentration on the Ion-Selective conduction in PVC/PEO blend as membranes in electrolyte electrodes, and she found that temperature, frequency and PEO content affect the dielectric behaviour of the blended membrane.

In the present study, the conduction process by ion exchange in a solid PEO/$MnCl_2$ membrane is investigated as a function of applied frequency and concentration. The main object of this study is giving information concerning the electrical behaviour of PEO/$MnCl_2$ composite by using the impedance spectroscopy which is one of the powerful techniques to characterize the dielectric properties as we reported in several previous publications [12,14]. Therefore, thin films based on PEO with $MnCl_2$ salt as a reinforcement filler were used in the present study .We believe that this study is of great interest for some applications in the electrical industry by using some blended polymeric membranes.

Experimental

Composite Preparation:

The resin used in this work is poly(ethylene oxide) of molecular weight(MW=5 millions) was obtained from CNR(Nopoli-Italy).Ordinarily, the salt $MnCl_2$ was ground into fine powder by Agate mortar and sieved by a U.S. standard sieve of size (63 μm).Polymeric thin films of thickness range(50-150)μm with different salt concentrations (0,5,10,15 and 20)wt.% were obtained.

All the polymer composite films were prepared by casting from solution (casting method). PEO powder was dissolved in a suitable solvent Tetrahydrofuran (THF) at (30) ^{o}C, also at the same time the salt ($MnCl_2$) was dissolved in (THF) and at the same temperature. Later, the solution of $MnCl_2$ was added to the dissolved polymer at a suitable viscosity. The solutions were mixed thoroughly for (4-6) hours by using a magnetic stirrer at room temperature until a homogenous solution is obtained.

Then the mixture was cast into a stainless steel ring resting on Teflon substratum and waiting for a few days until the solvents have evaporated. All the samples were dried in vacuum oven at 40^{o}C for two days. The drying process was repeated until prepared membranes have fixed weight to ensure the removal of solvent traces.

Impedance Measurements:

Impedance measurements were carried out using HP 4192A impedance analyzer. The real and imaginary parts of the complex dielectric constant were calculated from:

$$(1)\ \varepsilon` = \frac{Z_i}{2\pi f C_o Z^2}$$

$$\varepsilon`` = \frac{Z_r}{2\pi f C_o Z^2} \qquad (2)$$

Where f is the frequency, Co = (ε_o A/T) is the electrodes capacitance, A the area of the disk electrode, □□□ the permitivity of the free space, and T the specimen thickness of the membrane. The Impedance Z is given by ($Z=Z_r-jZ_i$), Where Z_r, Z_i are the real and the imaginary of the impedance, respectively. The AC electrical conductivity (σ_{AC}) was calculated from the relation:

$$\sigma_{AC}=2\pi f \varepsilon_o \varepsilon`` \qquad (3)$$

Results and Discussion

Frequency dependence

Impedance measurements were performed on composite membranes of different $MnCl_2$ salt concentrations at room temperature, and in the frequency range from 10Hz up to 13MHz. Figure (1) shows the phase angle (Φ) versus frequency (on a logarithmic scale) of the applied field at different concentrations of $MnCl_2$ salt. It was found that all the prepared specimens (thin films) have generally the same frequency effect. Also, it was observed that the phase angle is always negative for all the thin films of different salt concentrations; indicating that the system is capacitive and can be represented by parallel capacitive and resistive (RC) networks (15). At lower frequencies (less than 300 Hz), accumulation of ionic impurities, interfacial polarization at specimen-electrode interfaces, and space charges in bulk voids cause strong distortion [16]. The shift of phase angle value (Φ) towards higher negative values shows that the material becomes more capacitive than resistive at high frequencies. It shows the shift of (Φ) toward low negative values with the increase of the salt content indicates that the composites have become more resistive than capacitive. This may be attributed to the existence of leakage (impurity) current in the bulk composite, which would increase with salt content, or may be attributed to hopping of ions by electron emission tunneling effect throughout the salt grains facilitated by the decreasing of the interdistance between the particles or grains as the concentration is increased [17,18].

Figure (2) represents the dependence of impedance (per unit length) on frequency at room temperature for specimens of various PEO-based concentrations. At lower frequencies (less than 300 Hz), the impedance has high values; with the increase of frequency, the impedance decreases to minimum values. This behaviour is observed for most dielectric materials as Polystyrene, Epoxy and PVC. The high impedance values at low frequency may result from the space charge in specimens or due to some structure defects (phase boundaries and grain accumulations), in addition to the electrode polarization effect [36,38]. The specimens with high salt concentration show less dispersion effects, which may be related to the creation of conduction paths throughout the salt network in the bulk. On the other hand, at frequency above 300 Hz, the impedance drops very quickly to attain relatively constant values at frequency above 50 KHz. This rapid decrease of Z indicates the response of the bulk with the alternating electric field. This behaviour may be attributed to the reduction of the interfacial polarization effect, which may exist at the electrode-specimen surface or internally on the filter matrix interface [24]. It was found that the measured impedance at the low frequency below 1 kHz, decreases rapidly below 10 Wt. % of $MnCl_2$ concentration and slowly decreases above it at higher concentration. Above 10 kHz, the impedance shows a slight decrease with the increase of $MnCl_2$ concentration. This decrease in the impedance is due to both the increase of salt concentration and the decrease of hindrance of polymer matrix (14,39). On the other hand, the decrease in impedance indicates that the material becomes more conductive. This behaviour may be attributed to the increase in intrinsic ionic migration, which depends on the chemical structure of the material, and in case of PEO polymer it involves protonic migration where protons are removed from the PEO molecules and transported through the ethereal oxygen local segmental motions, leading to an increase in chain mobility (31). Thus, proton migration in PEO and ion exchange of Cl^{-} ion in $MnCl_2$ may lead to high electrical conduction in the composite membrane [13,33,34].

Cole-Cole plots are usually used as a successful tool to analyze the impedance and dielectric data of dielectric materials. We use it here to characterize the dielectric behaviour of the PEO-$MnCl_2$ composite [19]. A plot of the real part (Z_r) and the imaginary part (Z_i) of impedance for different salt concentrations is shown in Figure 3. It can be seen from this figure that the plots have certain shapes that characterize many dielectric solids. The Cole-Cole construction yields slightly inclined and distorted semicircles. The geometrical shape of the complex impedance plane plots indicates that the membrane cell is electrically equivalent to (RC) networks, which reduces to a pure resistance at both high and low frequencies [20]. Similar results were obtained by other ion-exchange electrodes [8,21,22,23]. Extrapolation of these circles would intersect the real part-axis at different Z_r values. The distance of the intersection from the origin represents the ohmic bulk resistance at infinite frequency [16]. Also it can be seen that the bulk ohmic resistance is reduced as the salt concentration is increased, which corresponds to the increase in the electrical conductivity. This may be related to a possible increase in the number of conduction paths created in the specimen in addition to a decrease in the width of the potential barriers within the bulk regions of high conductivity. Therefore, more charge carriers may be able to “hop” by tunneling, resulting in the observed decrease in the bulk resistance [24]. Some physical parameters can be estimated to shed some light on the conduction process-taking place in the given membrane. For example, the relaxation time (τ) was found by two methods, one of them is by locating the frequencies of maximum Z_i by using Figure (3) and the equation: $\omega_{max}.\tau=1$, where $\omega = 2\pi f$. The Cole-Cole plots were approximated to semicircles [25] to calculate (τ). The other methods for locating (τ) by plotting log Z_i verses log f, and log Z_r verses log f, and locating the intersection point. The intersection point determines the frequency at which ($Z_r = Z_i$), under these conditions, $\omega\tau = 1$ (8). The values of the relaxation time (τ) for each semicircle are calculated by this method, and they are included in the table (1). The variation of the relaxation time (τ) with the salt concentration is shown in figure (4), where the relaxation decreases with the increase of the salt content. Consequently, the conductivity increases, because the transport process would become more rapid due to the enhancement of ionic conduction, which increases with the increase of the salt content in the sample.

Figure (5) represents the dispersion of the dielectric constant (ε`) of the samples calculated from equation (1) with different PEO $MnCl_2$-salt concentrations. The general trend of the curves is toward the increase of ε` with salt concentration, similar to most conductive composites

[11,26,27]. It was observed that ε` of all of the composite samples is higher than the ε` of the pure (PEO). At frequencies below 300 Hz, ε` shows a sharp increase, with a rate of (f^{-1}) dependence. This may be associated with Maxwell-Wanger mechanism, especially the electrode polarization effect (28). At low frequencies, accumulation of ionic impurities, space charges, and formation of an electrode-specimen interface takes place. These effects cause a large and rapid increase in the value of ε` [9,28,29]. At frequency above 300 Hz, ε` decreases very slowly to attain a constant value of ε`. The general behaviour of ε`, verifying the fact that for polar materials as (PEO) and ($MnCl_2$), the initial value of ε` is high, but with a rate of (f^{-1}) dependence[12,30,31]. The behaviour of the dielectric loss(ε``) which was calculated from equation(2) against frequency is shown in Figure (6). At low frequencies (ε``) has a high value and then it starts to decrease at higher frequencies. The low-frequency dispersion in (ε``) is attributed to charge carries, which leads to large losses at low frequencies.

From the behaviour the dielectric constant (ε`) and the dielectric loss (ε``), one can observe a strong frequency dependence especially at low frequencies, which reflects the behaviour of the polar materials. It is clearly seen that both (ε`) and (ε``) increase with salt concentration and decrease with the frequency of the electric field and they have a high value at low frequencies and a low value at high frequencies. These results suggest that polar entities of the (PEO) are effectively operating under the electric field. This behaviour can be understood as follows: at low frequencies, the time interval required for the molecular dipoles of the (PEO) polymer to response to the applied electric field is sufficient. This enables these dipoles to follow the oscillating field, i.e., the orientation polarization is high, which leads to enhance the dielectric constant values. While at high frequencies, the time interval needed for the dipoles to response to the applied electric field is insufficient. Hence, the dipoles are unable to follow the rapid alternation of the oscillating field. In other words, the dipoles of the PEO polymer are able to rotate in the direction of the applied field at low frequencies, but at higher frequencies their rotations seems to be blocked in a particular direction, i.e., the orientation polarization drops down greatly and leads to very small value of (ε`)and (ε``) at high frequencies (11,23,32), which is similar to the behaviour for polar polymer and materials (33). However, the general dispersion behaviour of the field PEO films reflects the dielectric characteristics of the polar semi crystalline polymer [12,34,35], i.e. dipole rotation or polar polarization. This dielectric behaviour explains the increasing in the AC conductivity at high concentration [36].

The AC conductivity ($\sigma_{A.C}$) was calculated from the equation (3) and plotted versus frequency for specimens of different salt concentrations as shown in the Figure (7). It can be observed that ($\sigma_{A.C}$) for pure PEO increases slowly with frequency. But for the other composite samples of salt concentrations 5, 10, 15, 20 wt.%, the (σ) increases rapidly with the increase of frequency. Also it can be seen that at high frequencies the conductivity ($\sigma_{A.C}$) increases rapidly with the increase of frequency, these results support the well known fact that the bulk A.C conductivity is induced at high frequency range, as reported previously by many researches on different composite materials [11,23, 37]. Another possibility is that at high frequencies, the dielectric loss (ε``) is dominated by ionic conductivity produced from the increased electronic and ionic mobility of the existing impurities and more ions and charges are moved [9,26]. The observed induced conductivity at high frequencies locates the given composite in the semi conducting level of the electronic material.

The values of the tangent loss (tan δ) were calculated by using the equation (tan δ=ε``/ ε`). The behaviour of the (tan δ) as a function of frequency for different salt concentrations is shown in Figure (8) .The curves indicate that certain structural relaxation events take place in the bulk of the composite specimens. The figure shows high values of (tan δ) at low frequencies. However, (tan δ) exhibits some oscillatory behaviour that may be due to the structural relaxation processes, and the peak depends on the relaxation time of each specimen, and (tan δ) decreases with the increase of the frequency when ($\omega\tau>1$), and the (tan δ) decreases with the increase of the frequency when ($\omega\tau<1$). Also, it can be seen that the peak position appear to shift monotonically toward high frequencies, while the peak height increases with the increase of salt concentration. These characteristics may be understood through the flow of the charge carriers that exists in the composite bulk [24]. On the other hand, the increase of the salt content makes the composite difficult to polarize, and therefore, over heating the losses are enhanced. However, the observed behaviour seems to be justified through the proposed argument.

Concentration Dependence:

Figure (9) shows the behaviour of dielectric constant (ε`) with salt content (wt.%) at different frequencies. It was found that ε` for the 100 Hz increases rapidly with the increase of the salt content up to 10 wt.%. Above 10 wt.% to 15 wt.%, the dielectric constant is decreased with the increase of the salt concentration; but ε` starts to increase rapidly above 15 wt.% salt content. For the range frequency 300 Hz to 10 kHz, it was found that the dielectric constant increases as salt wt.% increases at all frequencies. However, at 100 kHz, the dielectric constant is hardly increases slightly. Also, as the $MnCl_2$ concentration increases, the low frequency dielectric dispersion effect becomes stronger due to ions diffusion in the membrane bulk. This dispersion is a dominant mechanism caused by conductivity enhancement due to the increase of the salt content [16]. The behaviour of the dielectric loss (ε``) as a function of salt wt.% at different frequencies is shown in Figure(10). It was observed that within the frequency range 100 Hz - 100 kHz, the dielectric loss increases with the increase of the salt content up to 10 wt.%. But from 10 wt.% to 15 wt.% the dielectric loss is nearly independent of salt concentration for all the frequencies. Above salt content 15wt.% the dielectric loss starts to increase rapidly again. At frequency 100 kHz, the dielectric loss remains nearly constant with the increase of the salt content.

The observed results of (ε`) and (ε``) indicate that up to 15 wt.% of salt in (PEO) acts as an intermolecular plasticizer and is able to penetrate the molecular bundles of polymer, leading to chain separation. However, at still higher percentage of salt structural defects, interfacial polarization along with the polarization of the constituent of the composite specimen starts playing a role, giving rise to an increase in ε` and ε`` [36,39]. The sharp increase in dielectric loss (ε``) above 15 Wt.% salt concentrations may be attributed to a particular process which takes place in the composite structure [19,29].

Figure (11) shows the behaviour of the A.C. conductivity, calculated from equation (3), as a function of salt concentration at different frequencies. It was observed that the conductivity is enhanced with the increase of salt content; many authors reported the same behaviour on different composite systems [10,11,23]. Generally, the observed enhancement in the dielectric constants and AC-electric conductivity with increasing of the salt content is attributed to the ionic interaction-taking place in the bulk of the double electrolyte solid membrane. The bulk effect creates an excess in the movable ions and charged particles, especially the impurities. This behaviour is physically consistent, since the $PEO/MnCl_2$ composite becomes more ionic with the increase of salt content.

Conclusion

The research work presented in this paper deals with the electrical properties of $PEO/MnCl_2$ composite. The electrical conductivity, dielectric behaviour and impedance of these polymeric membranes were studied as a function of $MnCl_2$ concentration and the applied electrical field frequency through the impedance technique. From the obtained results the following conclusions are drawn:

1. Frequency and $MnCl_2$ concentration affect on the electrical and dialectical behaviour of the composite membranes.
2. The phase angle takes always negative values for all of the samples at different salt concentrations and frequencies which indicate that the specimens can be represented by (RC) networks.
3. Both the impedance and the dielectric behaviour showed frequency dependence, explained on the basis of the interfacial (space charge) polarization, dipolar polarization and on the decrease of the hindrance of the polymer matrix.
4. The dielectric constant and dielectric loss of the composite membrane increases with the increase of $MnCl_2$ content.
5. The complex impedance plots exhibit different mechanisms operating in the bulk related to the relaxation times of the relaxations influenced by the frequency and salt content.
6. The peak value of (tan loss) is shifted to a higher frequency at higher $MnCl_2$ concentration.
7. The AC conductivity increases with the increase of frequency, $MnCl_2$ concentration due to enhancement of ionic conduction in the membrane bulk.

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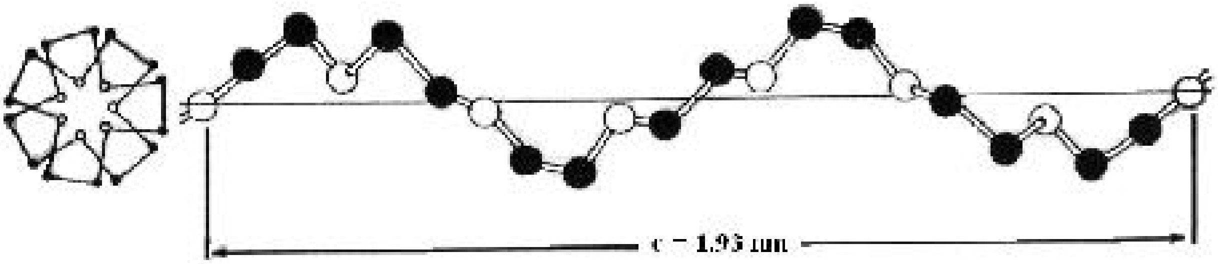

Picture (1): the molecule conformation of poly(ethylene oxide).
Where: ● represent the methylene group and ○ is the oxygen atom (4).

Table (1): Relaxation time as a function of $MnCl_2$ wt. %.

$MnCl_2$ (wt.%)	$\tau \times 10^{-6}$ (sec)
0	160
5	61.2
10	27.9
15	16.4
20	1.6

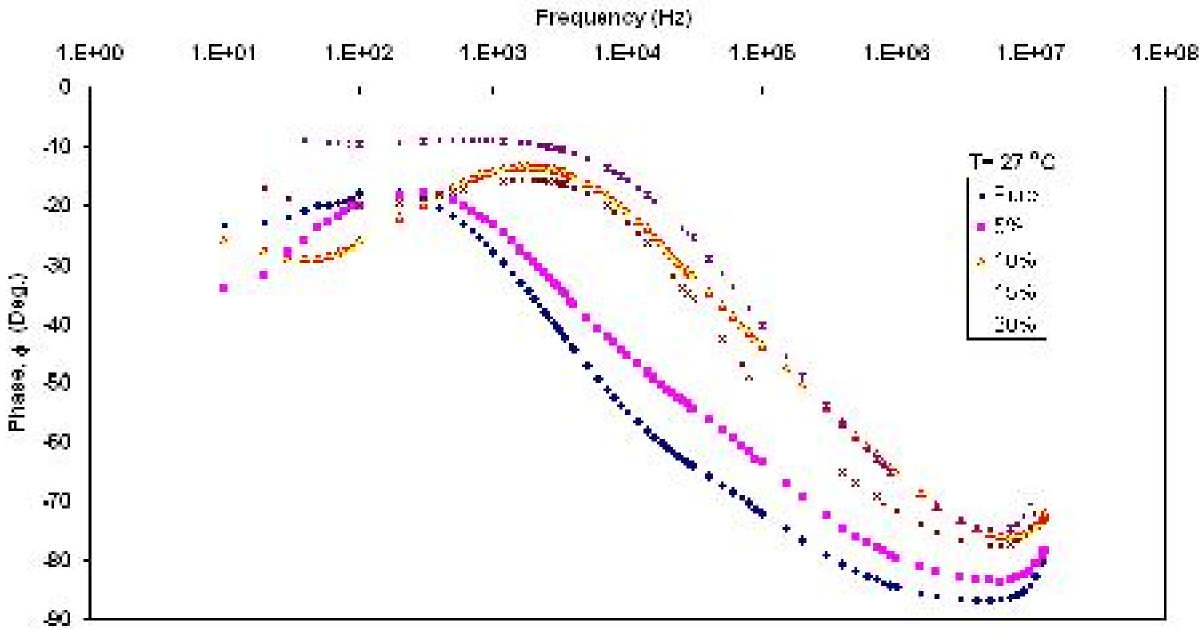

Fig. (1): Phase Angle Dependence on Frequency for Different Salt Concentrations.

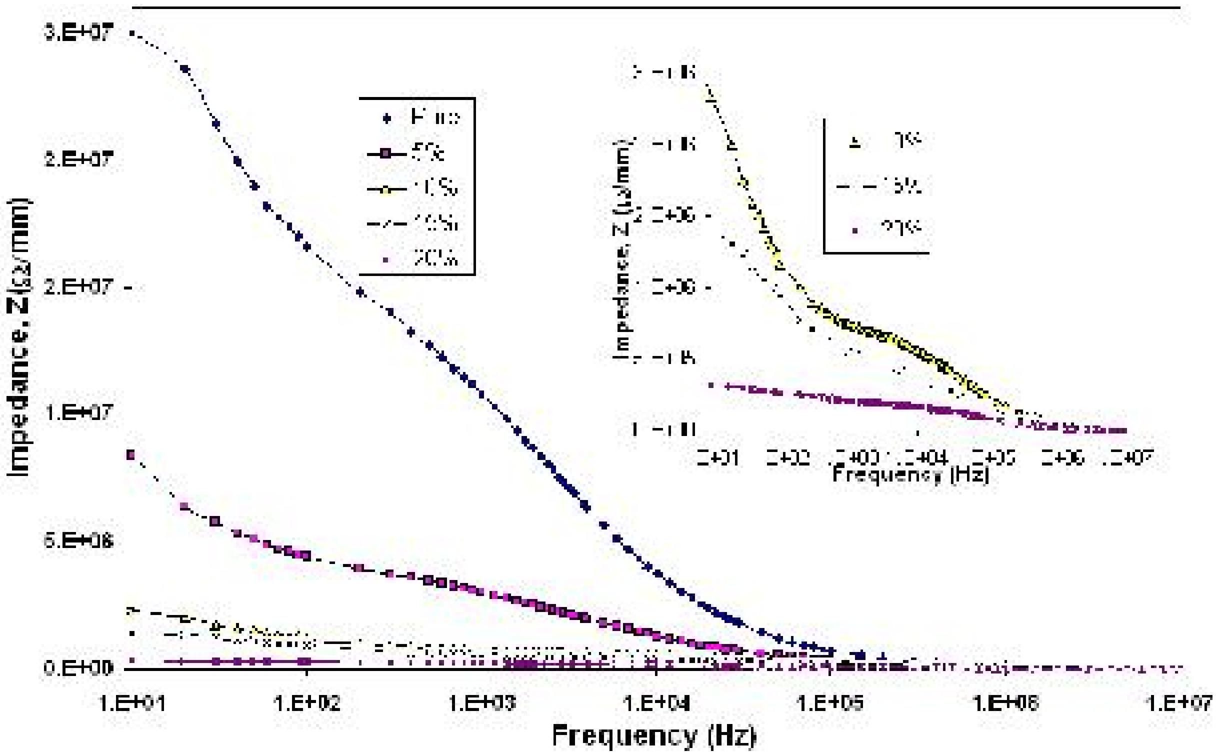

Fig.(2): Variation of the Impedance with Frequency for Different Salt Concentrations.

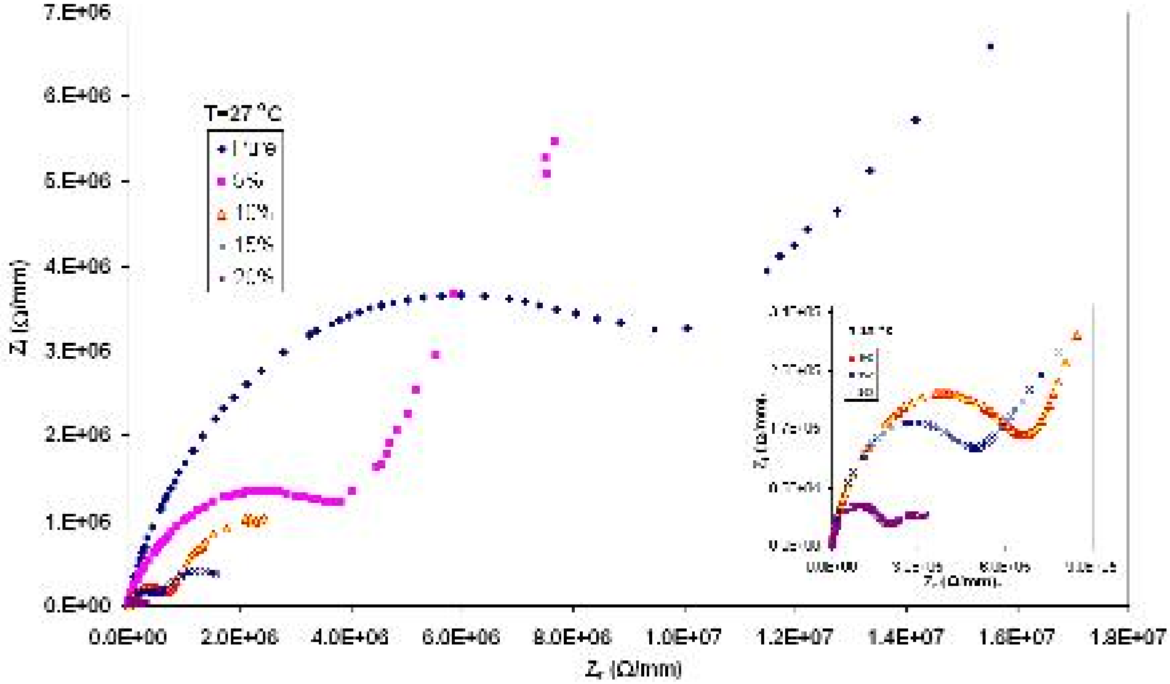

Fig. (3): Complex Impedance Plots for Different Concentration of (PEO/$MnCl_2$) Composite.

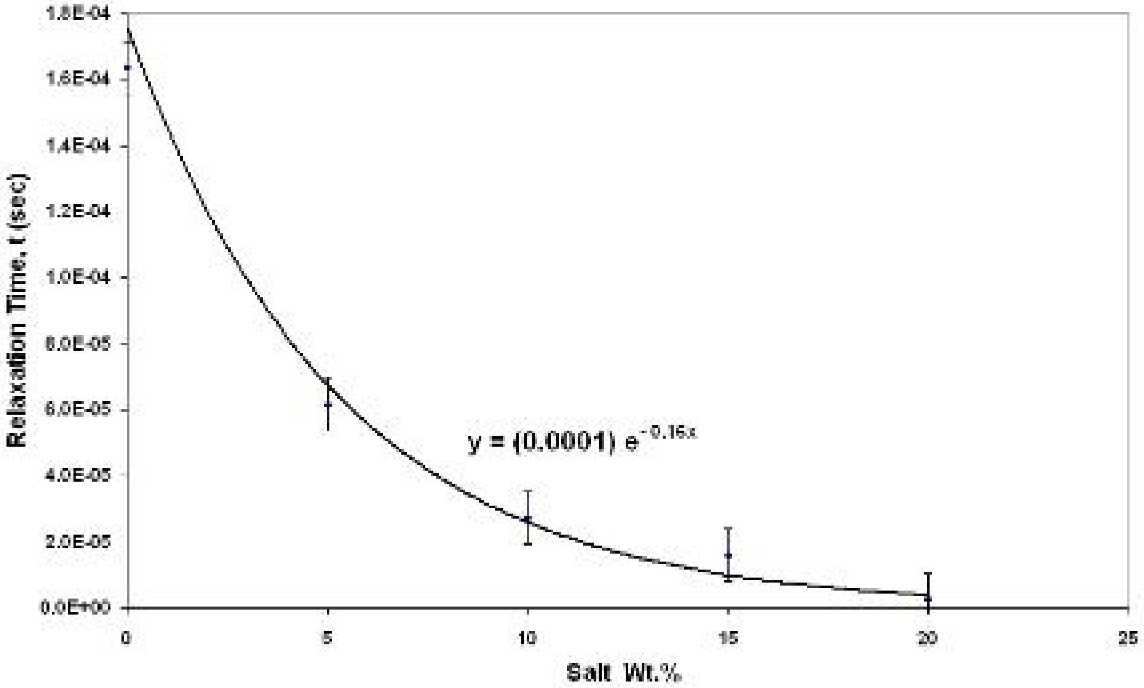

Fig. (4): The Relaxation Time as a function of Salt Concentration.

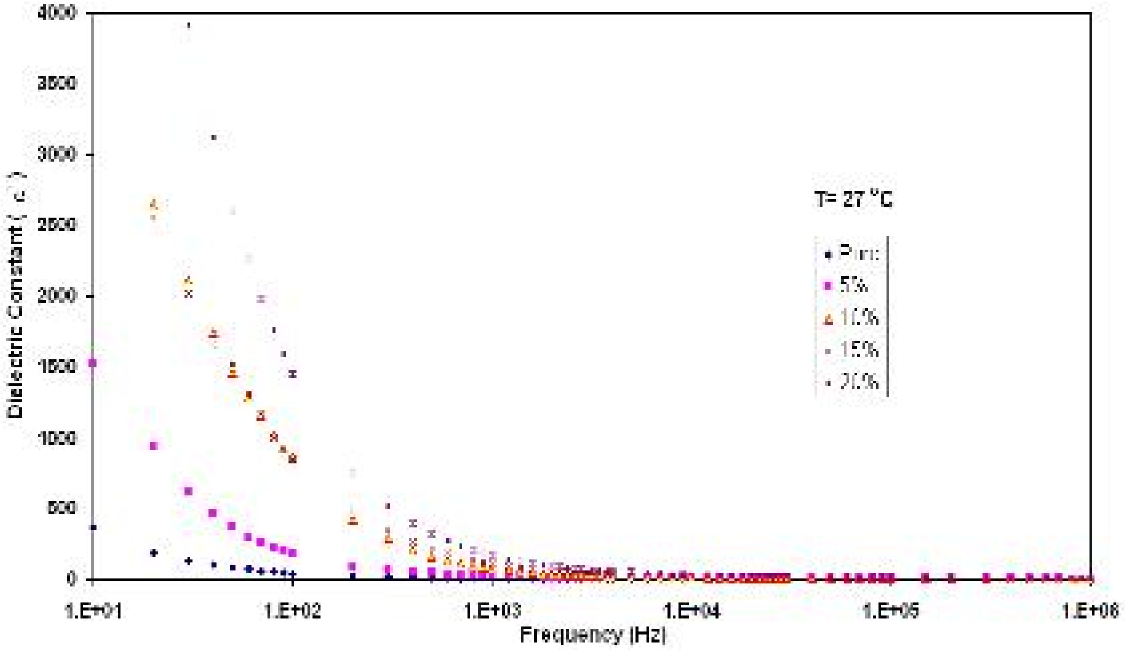

Fig.(5): The Dielectric Constant as a Function of Frequency for Different Salt Concentrations.

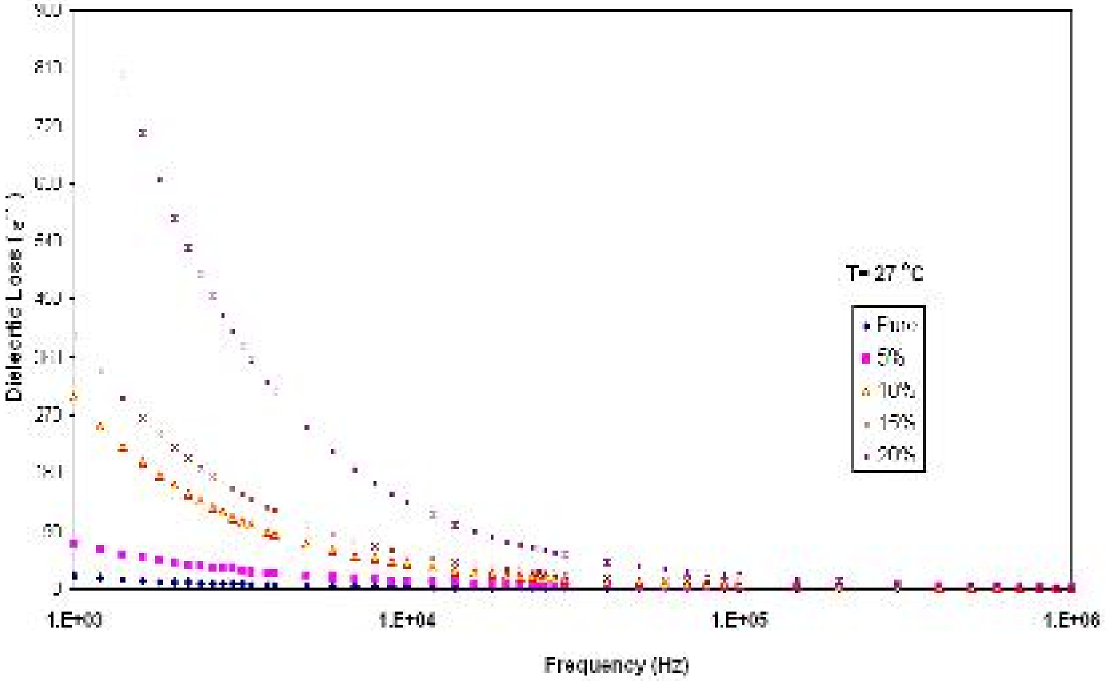

Fig. (6): The Variation of the Dielectric Loss as a Function of the Frequency for Different Salt Concentrations.

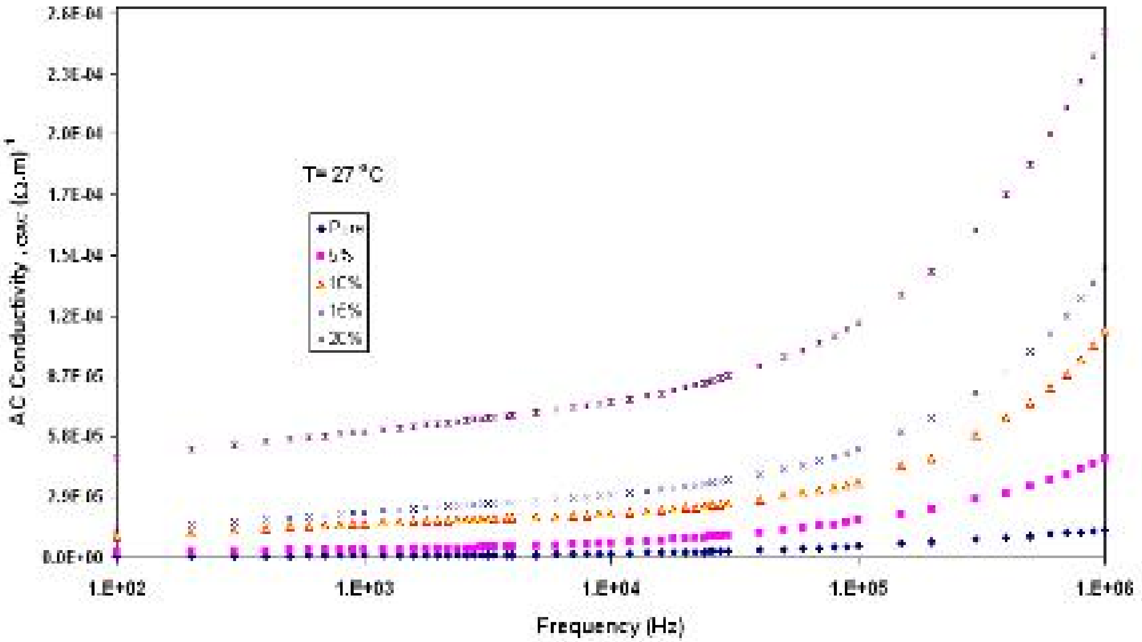

Fig. (7): The AC Conductivity of PEO as a Function of the Frequency for Different Salt Concentrations.

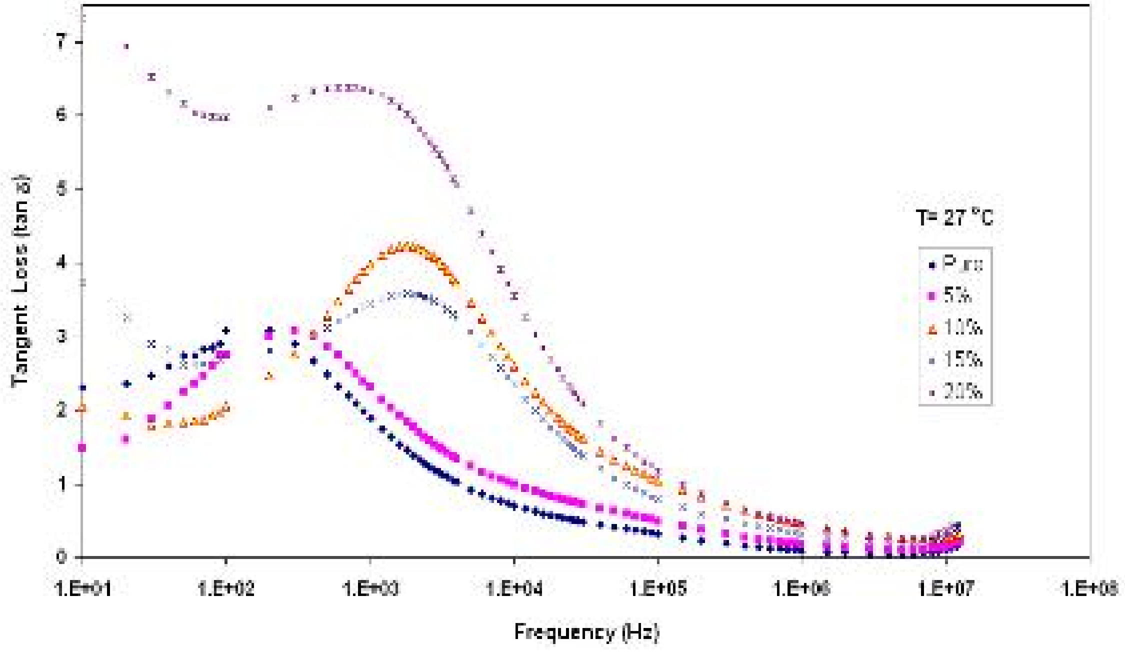

Fig. (8): The Variation of the Tangent Loss as a Function of the Frequency for Different Salt Concentrations.

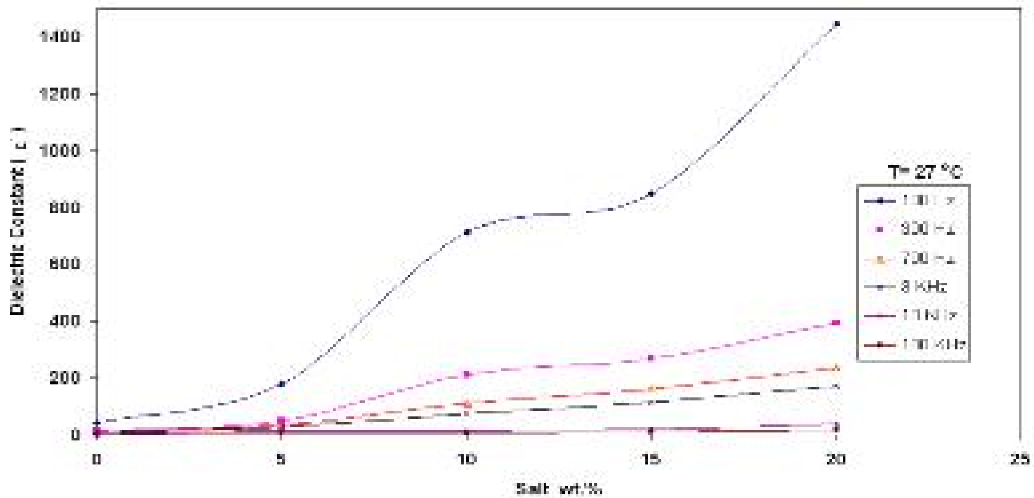

Fig. (9): The Dielectric Constant Behavior as a Function of Different Salt Concentration at Different Frequencies.

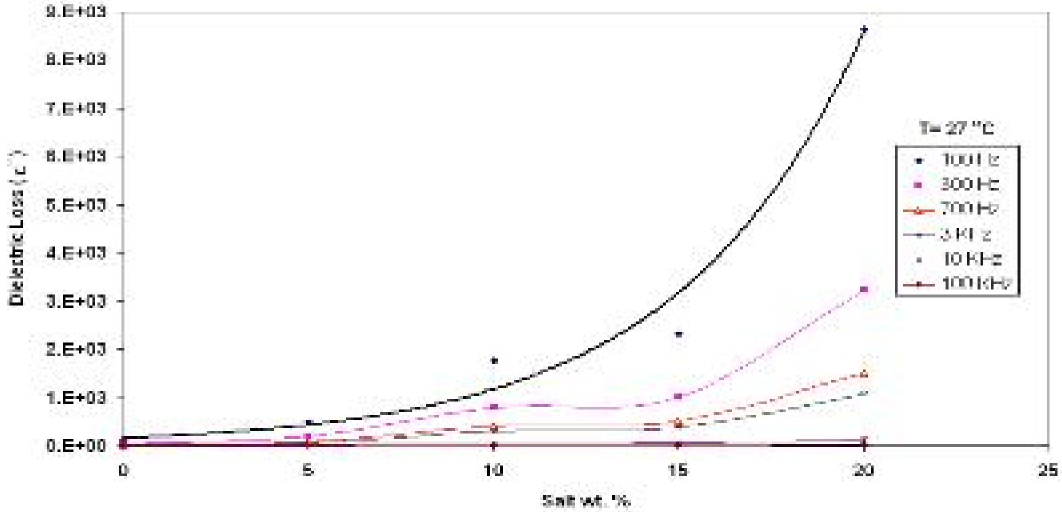

Fig. (10): The Dielectric Loss as a Function of Different Salt Concentration at Different Frequencies.

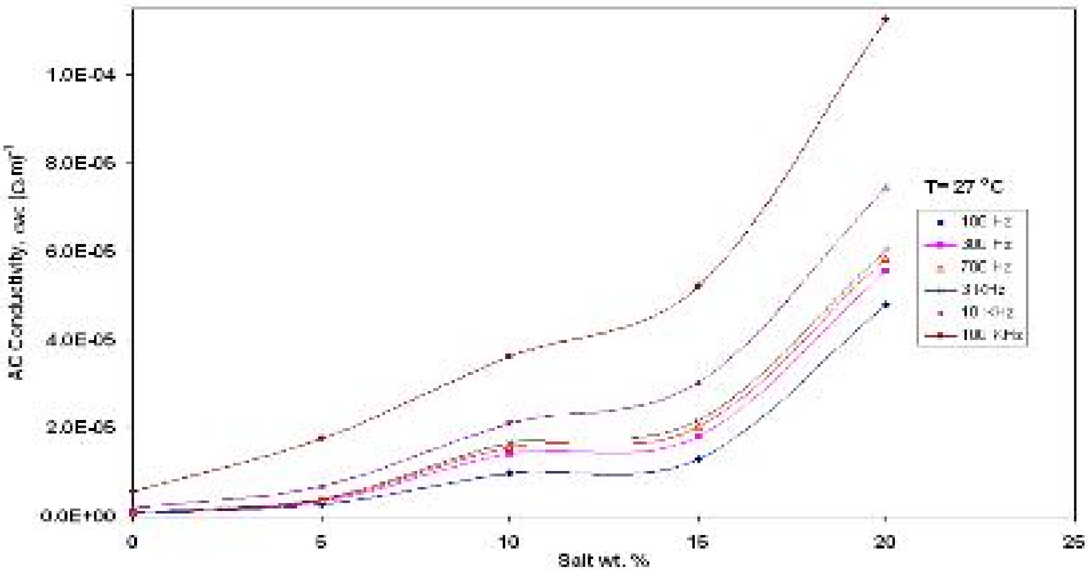

Fig. (11): The AC Conductivity Behavior as a Function of Different Salt Concentrations.

الخصائص الكهربائية للمبلمر بولي أوكسيد الإثيلين المطعم بالملح كلوريد المنغنيز

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الخلاصة

إن العمل المقدم في هذا البحث هو محاولة لدراسة الخصائص الكهربائية لمبلمر متراكب من المبلمر (بولي أوكسيد الأثيلين) كقالب ومن ملح (كلوريد المنغنيز) كحشوة و ذات تراكيز مختلفة داخل القالب بالمدى 0% إلى 20% . وقد تمت دراسة سلوك الممانعة الكهربائية وثابت العزل الكهربائي ومعامل الفقد و زمن الاسترخاء لهذا المتراكب في نطاق التردد(10هرتز –13 ميكا هرتز). وقد درس في هذا البحث تأثير تراكيز الملح وتردد المجال الكهربائي على الموصلية الكهربائية المتناوبة.

ولقد وجد من النتائج بأن الملح يعزز و يزيد التوصيلية الكهربائية، وبعد حساب ثابت العزل الكهربائي وجد أنه يزداد بزيادة تركيز الملح المستخدم، وقد لوحظ إعتماد ثابتي العزل والممانعة الكهربائية على التردد، وقد يعزى ذلك لبعض ظواهر الإستقطاب المعقدة وللقطبية العالية لهذا المبلمر. فزيادة تركيز الملح والتردد المسلط على العينة يؤدي إلى نقصان زمن الإسترخاء للمتراكب المحضر مختبرياً لتأثير الحركة الأيونية و وجود الشوائب في المتراكب.