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CHARACTERIZATION OF THE ELECTRIC PROPERTIES OF PEO/ALUM COMPOSITE DOPANT WITH CARBON BLACK NANOPARTICLES AT T=40 ^{o}C

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ABSTRACT

Electrical properties of the prepared polymer films, made of poly (ethylene oxide)(PEO) filled with electrolyte alum salt of different concentrations, and doped with conductive carbon black (CB) nanoparticles (0.1 wt%), have been investigated. Electrical properties were studied as a function of filler content and applied field frequency at T=40 ^{0}C. The observed physical constants of the casted thin films like AC conductivity, phase angle, impedance, dielectric constant, and electric modulus were determined. It was found that these measured quantities vary with the alum content and applied field frequency. The AC conductivity (σ_{ac}) increases with increasing alum concentration and frequency. The dielectric constant (ε') and the dielectric loss (ε'') of the composites decrease with frequency and increase with alum concentration. The dependence of the electric modulus on frequency exhibit a relaxation peak occurs at 500 kHz.

Keywords: PEO, CB, AC conductivity, dielectric, electric modulus.

1. INTRODUCTION

Electrical properties of polymer composites are important in many industries such as automotive, aerospace, marine, building products, packaging and consumer goods. Electrical tests, in general, are measurements of the conductivity, resistance or charge storage on the surface or through the plastic material. Electrical conductivity or specific conductivity is define as a measure of a material's ability to conduct an electric current. When an electrical potential difference is placed across a conductor, its movable charges flow, giving increase to an electric current. Solid polymer electrolytes (SPE) still promising materials for electrochemical and other device applications. They are made of a solid polymer dispersed with an electrolytic ionic substance to form ionic systems where the electricity is transported under the applied electrical field by mainly ions diffusion. Recently, they are successfully used in high energy density rechargeable batteries, full cells integrated optical devices, and electrochromic displays.

Electrical properties of polymers can be designed for a specific requirement by addition of convenient dopant substances or conducting elements. Poly (ethylene oxide) (PEO) has electrical insulation properties, it has a low melting point (T_m= 60 ^{0}C), low glass transition temperature (-65 ^{o}C) which allows transport the ion at surrounding temperature and low toxicity. Poly (ethylene oxide) is the best polymer used, because its good process ability, large solvating power with ions and outstanding mechanical properties. Due to its low cost and easy production, it is often used for commercial purposes. PEO has a property to form molecular complexes which enhances the electrical conductivity [1-3].

Filler materials are most often added to polymers to improve tensile and compressive strengths, toughness ,abrasion resistance, thermal stability and dimensional, and other properties. Polymers that contain fillers may also be classified as composite materials. Often the fillers are cheap materials that replace some volume of the more expensive polymer, reducing the cost of the final product [4].

The alum filler can be found in form of glassy transparent crystals and soluble in water. It is a solid electrolytic ionic salt results from hydration of sulfate of a singly cation (e.g., K^{+1} and the sulfate of any one of triply charged Al^{+3}) to form alums with sulfates of singly cations of potassium and other elements. It is used commonly in water purification, dyeing, fireproof textiles and leather tanning. The addition of electrolytic filler greatly enhances the ionic conductivity and affects the bulk properties of the composite. The interactions of an alkaline ion with a polymer matrix determine its possible application in energy batteries and other solid state electrochemical devices. The ion conduction in the composite bulk was mainly attributed to ionic interaction and impurity activity taking place during the passage of a current into the composite.

One of the most common conductive fillers is Carbon black nanoparticles (CB). When it doped in polymers, may reside at various sites, it may go substitution-ally into the polymer chains and composed charge transfer complexes (CTC), or may exist in the form of molecular combination between the polymer chains [5]. CB is a non-crystalline form of carbon; it is conductive and a lot composed of carbon atoms or groups of nearly spherical shape. The most important purpose of CB used as filler in polymers to import thermal and electrical conductions, i.e., conductive polymer composites [6-9].

The electrical, thermal, optical, and mechanical characterization is an essential for the industrial development of thin films of new polymers, composites and advanced materials that can be used as optical devices, polarizers filters, total reflectors, and narrow pass-band filters [10].

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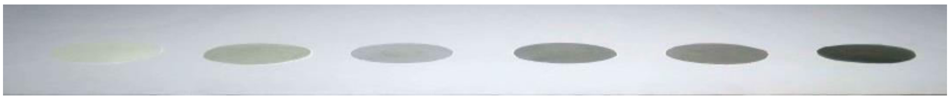
In the present comprehensive paper the studied PEO thin films are doped with CB nanoparticles at T=40 °C. Also, this paper covers electrical conductivity measurements and temperature effect T=40 °C on few physical parameters which focus on enlarged characterization and provide deeper understanding to energy transport processes occur in doped thin films. The AC electrical conductivity is studied as a function of frequency in the range (3 kHz - 5 MHz) at T=40 °C. A number of measured quantities as dielectric constant, phase angle, impedance, and AC conductivity were determined.

2. EXPERIMENTAL WORK

2.1 Materials and composites thin films preparation

The potassium aluminum sulfate filler was a glassy transparent crystals and soluble in water. This solid alum was ground into a fine powder of average particle size 18 µm. The powder kept in an oven overnight at 40 °C to decrease the water content. The electrolyte alum filler has a chemical formula ($KAl(SO_4)_2 \cdot 12\,H_2O$), it is one of series of crystallized double sulfates include some impurities. Poly (ethylene oxide) has average molecular weight of 300,000 g/mol, it was used to prepare composite films of PEO/alum dopant with carbon black nanoparticles by casting from solution made of PEO and alum. Poly (ethylene oxide) powder, alum powder and carbon black powder were mixed together and dissolved in methanol as appropriate solvent. The solution was then stirred continuously by a rotary magnet at room temperature for three days until the mixture appear in a homogeneous viscous molten appearance. The mixture was immediately casted to thin films on to a glass mould (plate). The methanol was allowed to evaporate completely at room temperature by waiting for two days under atmospheric pressure. All the samples were dried in the oven at 40º C for two days. The thickness of the thin films composites are measured by a digital vernier caliper. Least division of the device is 0.001mm. The thicknesses of all films are measured at 5 various places, chosen randomly selected, and then average of it is taken in the count. The obtained average value of thickness approximately is 100 µm. The color of thin films gradually changes with higher alum concentration as Figure-1.



Pure PEO 16 wt.% alum 12 wt.% alum 8 wt.% alum 4 wt.% alum 2 wt.% alum

Figure-1. Appearance of PEO/alum composites doped with CB.

2.2 Electrical measurements

The AC electric properties of the PEO/Alum composite dopant with carbon black were studied through measurements of the impedance (Z), and the phase shift angle (φ) by using Hewlett Packard (HP) 4192A impedance analyzer. The test specimens were placed between two copper electrodes in a sample holder. These electrodes were connected through cables to the impedance analyzer. Impedance measurements were performed in a frequency range from about 100 kHz up to 3 MHz Under the effect of temperature (40ºC). To study the temperature effect on the impedance and the phase angle, the sample holder was placed in oven chamber. Accurate temperature readings were taken in a steady state condition by a thermocouple inserted beside the test specimen in addition to the temperature readings of the oven dial. The temperature readings were taken below the melting temperature of PEO which is about 60 °C.

3. RESULTS AND DISCUSSIONS

The alum filler appended to the matrix of polyethylene oxide to form solid electrolyte thin films is being searched to evaluate the role of the filler in the process of the ionic conduction when the electric field is affected. The purpose of studying electrical properties in polymers is to realize and type and nature of the charge transmission in conducting materials [11-12].

Some theoretical relations are required to calculate the electrical properties of PEO/alum composite with carbon black dopant under various cases as: concentration of filler, effective frequency of electric field, and temperature.

Dielectric materials are electric insulators that can be polarized by an applied electric field. When a dielectrics is placed in an electric field, electric charges do not flow through the material, as in a conductors, but only slightly shift from their average equilibrium positions causing dielectric polarization. Layers of such substances are commonly inserted into capacitors to improve their performance, and the term dielectric refers specifically to this application.

Connecting a resistor (R) to a capacitor (C) in parallel, the impedance (Z), the real component of the impedance (Z') and the imaginary component of the impedance (Z'') of the circuit are

$$Z = \frac{R\,(1 - i\omega\,C\,R)}{1 + (\omega\,C\,R)^2} \tag{1}$$

$$Z' = \frac{R}{1 + (\omega\,C\,R)^2} \tag{2}$$

$$Z'' = \frac{\omega CR^2}{1+(\omega CR)^2} \tag{3}$$

The dielectric constant (ε') which is related to the stored energy within the medium, and the dielectric loss (ε'') which is related to the loss of energy within the medium in form of heat generated by an electric field are determined from these relations [13].

$$\varepsilon' = \frac{Z''}{2\pi f C_o Z^2} \tag{4}$$

$$\varepsilon'' = \frac{Z'}{2\pi f C_o Z^2} \tag{5}$$

Where C_0 is the capacitance without the thin film, and f is the frequency (AC) of electric field

The AC conductivity (σ_{AC}) of the thin film is given by the relation

$$\sigma_{AC} = 2\pi f \varepsilon_0 \varepsilon'' \tag{6}$$

3.1 The effect of the field frequency on the electrical properties

In the range of frequency from 30 kHz up to 5 MHz, impedance measurements have been carried out on polyethylene oxide/alum thin films doped with carbon black nanoparticles. Figure-2 displays the relation of field frequency with phase angle (φ) (phase difference between the used voltage and current) for thin films of various concentrations of alum and pure (PEO) at (T=40°C). Always (φ) is a negative quantity, denoting that the bulk material can be created of resistive and capacitive connections. Figure-2 displays the change of (φ) across minimum negative values with increasing alum concentration, denoting that the resistivity will be more than capacity of the thin films due to addition of ionic and electronic dopants.

Figure-3 displays the difference impedance (Z) per unit thickness as a function of frequency at (T=40°C). This figure displays that the impedance decreases with increasing frequency. At low frequency, the high (Z) values are due to effects of space charge polarization in thin films, electrode polarization, and several structural defects (phase boundaries, grain accumulations) [14-15]. The rapid decrease of impedance values shows a response of the bulk to alternating applied electric field. This behavior may be referred to decreasing of the effect of interfacial polarization, which may found at the surfaces of electrode-sample [16-17]. The Impendence decreases with increasing the frequency due to the polarization effects, formation of connected carbon black paths and the enhancement of electronic mobility.

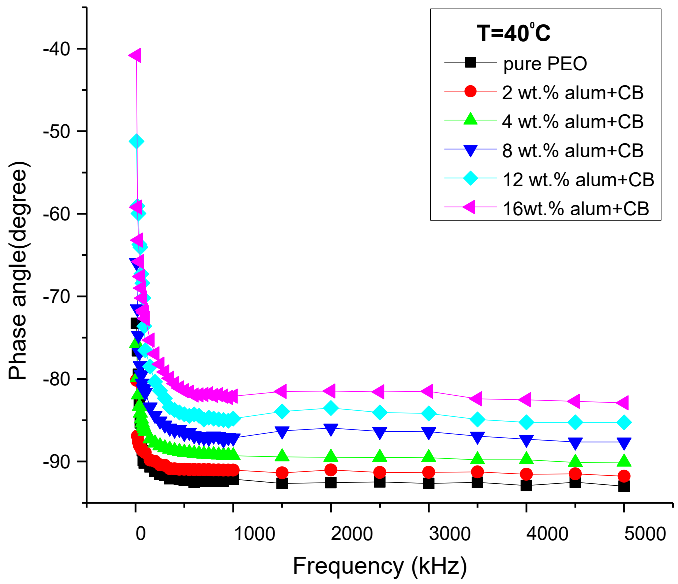


Figure-2. Phase angle versus frequency for PEO/alum samples.

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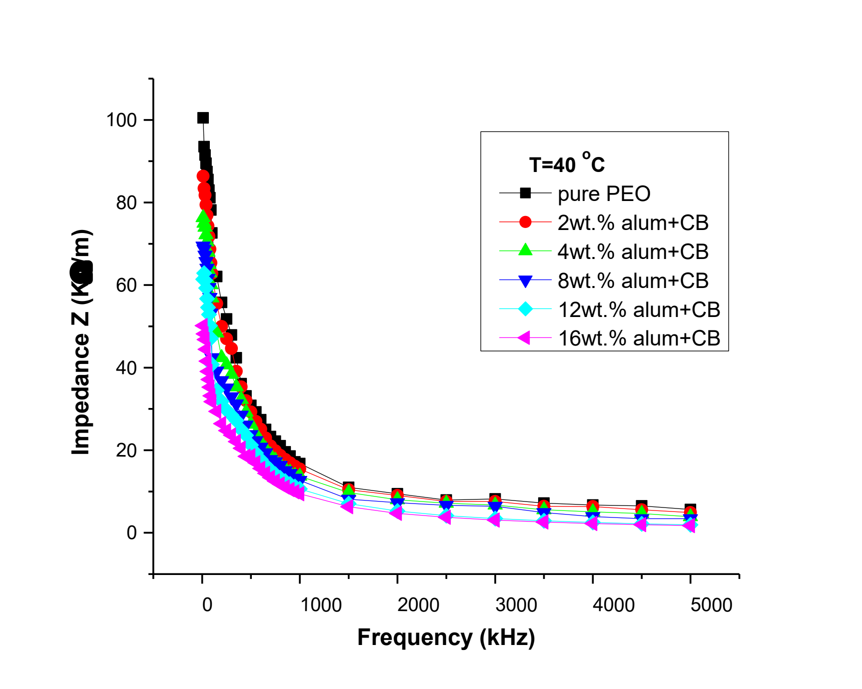

Figure-3. The variation of impedance with frequency for PEO/alum samples.

In Figure-4, with increasing frequency the observed decrease of the dielectric constant (ε') may be illustrated on the fundament of space charge polarization (Wagner-Maxwell effect) which slightly repairs and promotes for this behavior occurred by the orientational polarization. The dielectric constant of pure PEO is lower than that of composite samples. The noticed progress in the PEO insulation property is referred to electrons and charge complexes incorporated in the alum bulk and the conductive carbon black dopants, respectively, as seen from increasing the alum concentration with the increase of the dielectric constant.

Figure-5 displays the behavior of the dielectric loss (ε'') at (T=40°C), it has a high value at low frequencies, after that at high frequencies it begins to decrease. The low frequency dispersion in (ε'') is referred to the charge carriers (carrying electrical charge as electrons, holes, and ions, particles move freely), the ions (K^{+1} and Al^{3+}) are charge carriers found in alum, and the charge carriers in carbon black are electrons, which cause large losses at lower frequencies, and to increase polarization influence [18].

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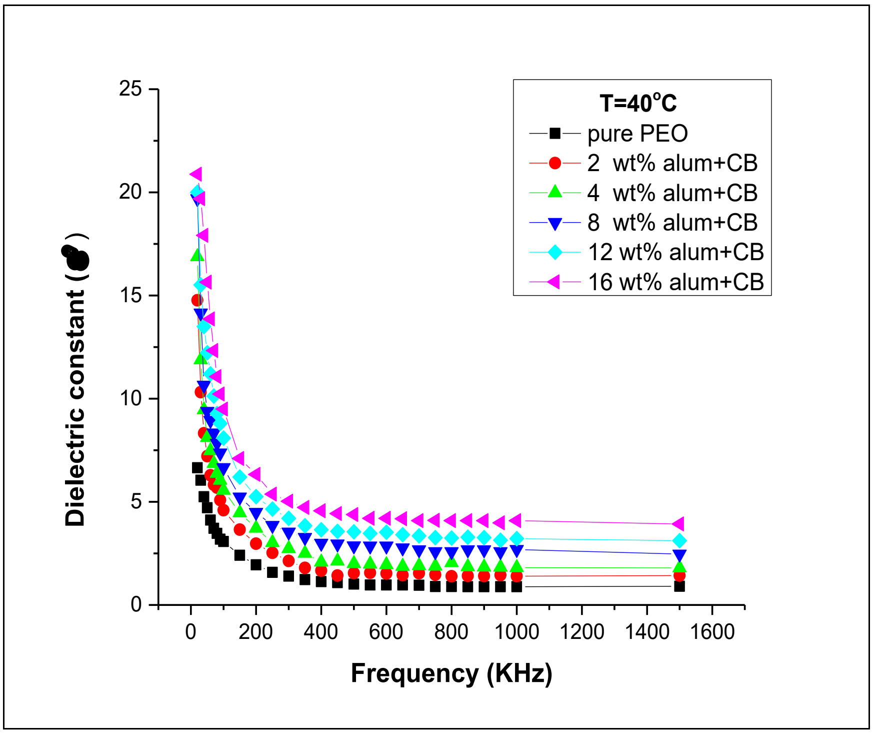

Figure-4. Dielectric constant versus frequency for alum/PEO composite

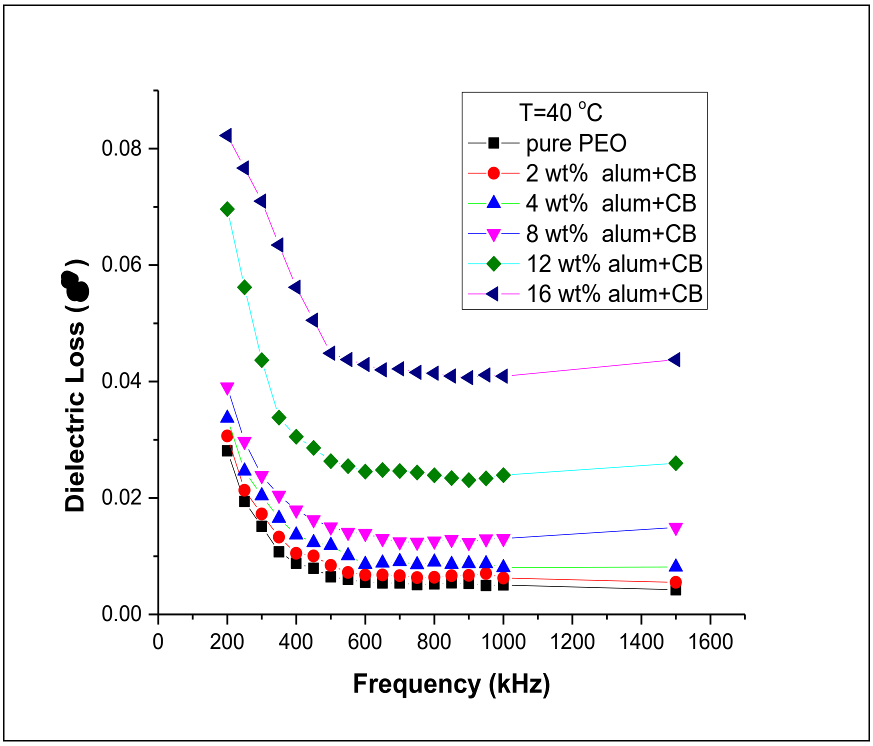

Figure-5. Dielectric loss versus frequency for alum/PEO composite.

From (ε') and (ε''), specifically at low frequency, a frequency dependence is observed, which represents the behavior of the polar materials as alum [19]. The polarity of alum is due to difference in electronegativity between aluminum and potassium, and the geometry of alum is octahedral. It is clearly observed that both (ε') and (ε'') decreases with the electric field frequency. These results propose that polar substances of the alum and PEO polymer of the polar semicrystalline polymers i.e., dipole rotation or polar polarization, the PEO/alum and dopant carbon black composites are effectively operating under the high electric field.

Figure-6 shows that with increasing the frequency and alum concentration, the AC conductivity

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(σ_{AC}) value increases, because more ions and charges can move at higher field, which causes increase of the ionic conduction process. This result prove that at high frequency the bulk AC conductivity (σ_{AC}) is induced [20-21].In ionic materials, cation vacancies allow ionic motion in the direction of an applied electric field, this is referred to as ionic conduction. It can be deduced that the ionic conduction is promoted at higher values of alum content and frequency. The electronic and ionic interactions in the bulk of the polymer electrolyte cause the increasing in the AC-conductivity and dielectric constants. A surplus in movable ions and charged particles are created by the bulk effect, especially the impurities [22].

The dopant carbon black particles form continuous paths in the polymer matrix. The free electrons move through these continuous paths from end one to the other under the applied electric field. This movement causes the process of electrical conduction based on the well-known conduction path theory [23]. The AC-conductivity increases at high frequencies and this is predictable as at higher field extra charge carries can move, resulting enhancement of conduction mechanism. The observed induced conductivity at high frequencies locates the given composite in the semiconducting level of the electronic materials [24].

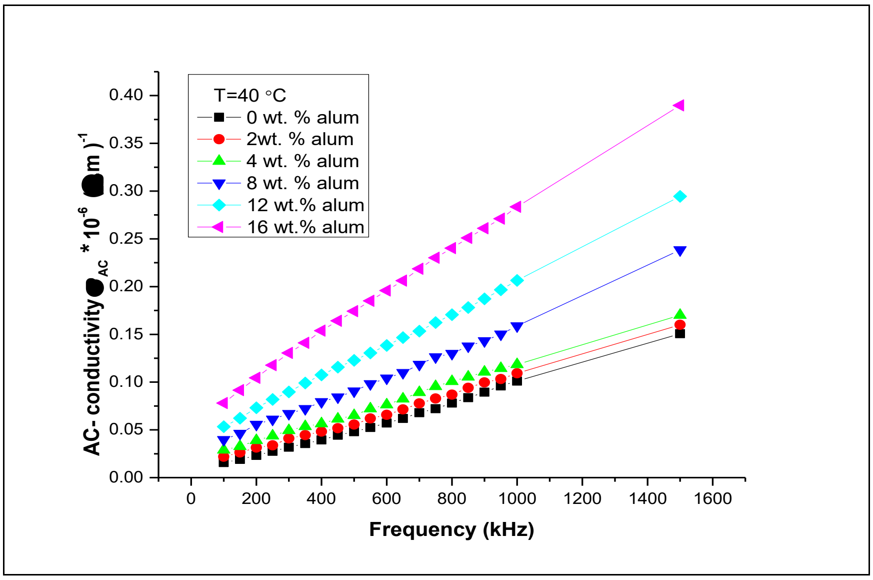

Figure-6. Dependence of AC conductivity as a function of frequency.

3.2 The relation between alum concentration and electrical properties

Figure-7 shows that, at different frequencies, impedance decreases when the alum filler substance increases at temperature 40°C. This decreasing in (Z) refers to free electrons and ions transfer, due to impurities in the alum and carbon black dopant. The electrical conduction increases because of these reasons.

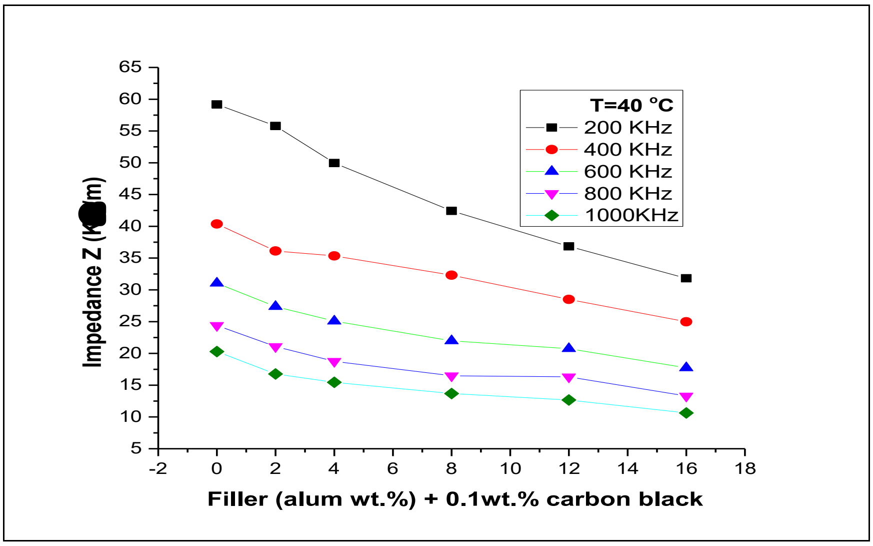

Figure-7. Variation of impedance with alum concentrations.

Figure-8 displays that with increasing the frequency, the dielectric constant (ε') decreases, while it increases greatly from about 0.84 for neat polyethylene oxide to 6.3, with increasing the alum concentration up to 16wt.%. The high increment in (ε') with concentration of alum is mainly due to the contribution of ions and free electrons. Carbon black particles produce conductive paths which make the composite more conductive, resulting enhancement of (ε'). The conduction attitude of the composites is controlled by the content of the conduction filler [25]. A similar behavior of these composites was observed for the dielectric loss (ε'') as shown in Figure-9. The increase of (ε'') may referred to the interfacial polarization of such a heterogeneous system [26].

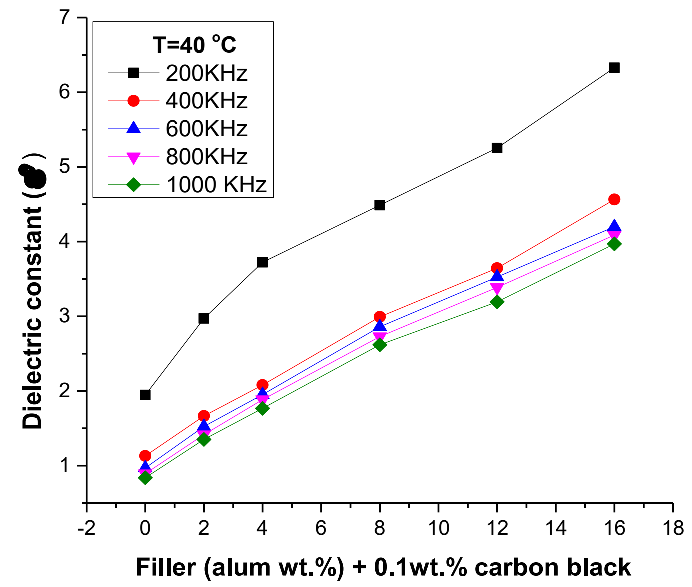

Figure-8. The dielectric constant versus alum concentrations.

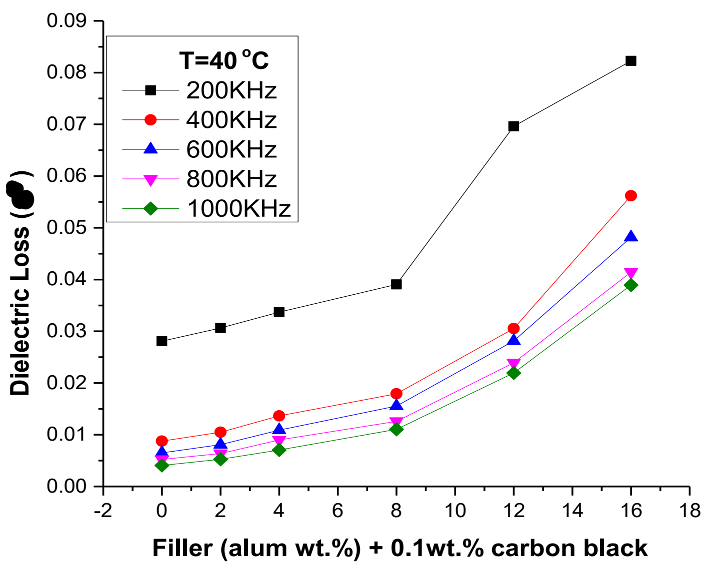

Figure-9. The dielectric loss versus alum concentrations.

Figure-10 displays that at different frequencies, the (AC) conductivity (σ_{AC}) increases with increasing filler content, which means with increasing of the conducting filler content the resistivity of polyethylene

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oxide/alum composites decreases. The increase in ionic conductivity with the alum content is referred to increasing of the concentration. The motion of ions in solid polymer electrolyte is liquid like mechanism while the ions movement through the polymer matrix is supported by the high amplitude of the polyethylene oxide segmental motion. The segmental mobility is lower in the crystalline region than in the amorphous region of the polymer chains. Accordingly, the continual increase in polymer conductivity may have been due to reduction in the crystallinity degree during the casting process, or increasing in the amorphism [27]. The ionic size is one factor in decreasing ions mobility that lead to increasing polymer conductivity. Since K^{+1} and Al^{3+} are rapid conducting ions in some crystalline and amorphous materials, its combination in a polymer would promote there's electrical and optical behavior [28].

Carbon black particles become interconnected and create infinite continuous paths within the PEO matrix, which allows a current to flow through the composite thin film. Adding more carbon black particles results in an increasing in the number of conductive paths, and a conductive network is formed [29].

The increase in (AC) electrical conductivity at low CB particles concentrations, below threshold value, results from electrons that effectively tunnel between isolated carbon black particles domains with diminishing the separation distances [30]. Increasing the carbon black particles content, the conductive paths become larger and spaces between adjacent clusters are diminishing [31]. The (AC) conductivity value increases because of carbon black particles have higher value of dielectric constant also because of the interaction between the particles of carbon black which will increase and will create more conductive network in the PEO matrix as carbon black content increases [32]. When carbon black particles are doped in the composite the gap between the particles diminishes and conductivity increases because the charges transport gets easier [33]. This leads to an increase of the AC conductivity by means of formation of per- collating paths and electron conduction.

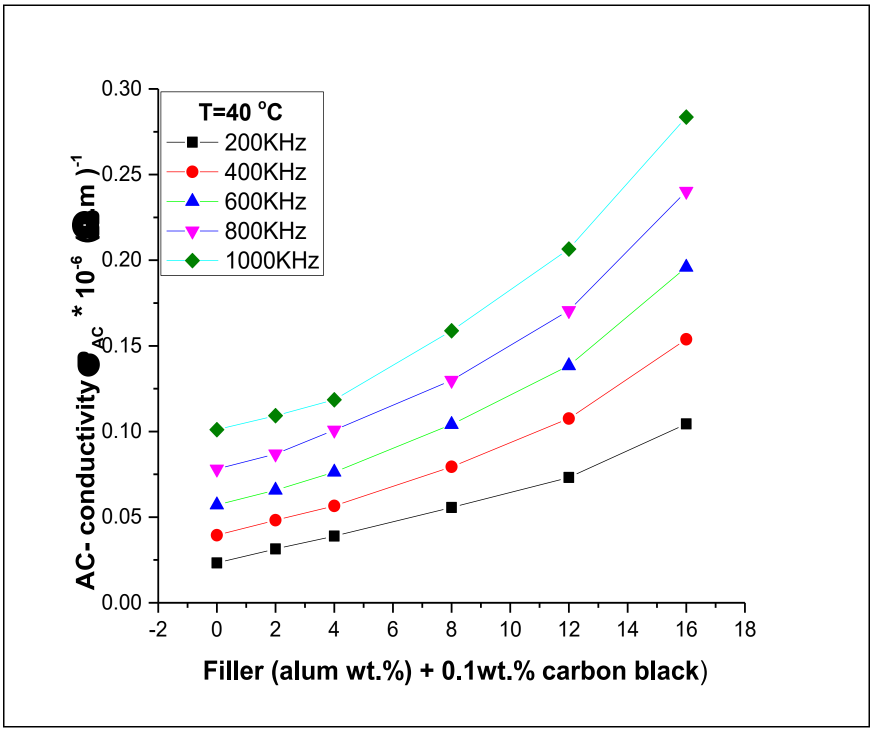

Figure-10. AC conductivity versus alum concentrations.

It was found that AC-conductivity values increase Under the effect of temperature (40ºC) at all frequencies, and this is due to electron and impurity activation that increases with temperature, and to ionic and molecular mobility of PEO polymer stimulated at high temperatures, i.e., the flow of electrons or charged ions are established between the polymer chains and thus leading to higher electrical conduction which is relatively similar to ionic and semiconducting materials. Also the increasing in the conductivity with temperature can be interpreted in terms of a hopping mechanism between local structure relaxation, coordination sites, and segmental motion of the polymer. When the amorphous region in the PEO structure increases, the polymer chain acquires faster internal lattice modes in which bond rotations produce segmental motion. This, in turn, favors the inter chain hopping movements, and the thus conductivity of polymer becomes high. Also, increasing the alum concentration with more charge carriers result in increasing structure defects, which can create new energy levels inside the forbidden energy gap at high temperature. Increasing temperature can increase

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the movement of carbon black nanoparticles in the thin films bulk that are trying to create paths network [34-35].

3.3 Electric modulus

The electric modulus formalism is used to study electrical relaxation phenomena in many polymers and vitreous ionic conductors. The physical nature of the electric modulus is used to make a correlation between the conductivity and the relaxation of ions in the materials. This approach can effectively be employed to study bulk electrical behavior of the moderately conducting samples.
If the dielectrics have mobile charges, then it seems convenient to concentrate on the electric field's relaxation under the constraint of a constant displacement vector, which performs to the inverse dielectric permittivity and the definition of electric modulus. The utility of using the electric modulus to interpret bulk relaxation properties is that variations in the large values of permittivity and conductivity at low frequencies are minimized [36]. The electric modulus is a good tool to reveal any structural relaxation takes place under the effect of applied field frequency.

The complex electric modulus (M*) is defined in terms of the complex dielectric constant (ε*) and is represented as:

$$M^* = \frac{1}{\varepsilon^*} = \frac{1}{\varepsilon^` - j\varepsilon^{``}} = \frac{\varepsilon^`}{\varepsilon^{`2} + \varepsilon^{``2}} + j\frac{\varepsilon^{``}}{\varepsilon^{`2} + \varepsilon^{``2}} \quad (7)$$

$$= M^` + jM^{``},\ j = (-1)^{1/2} \quad (8)$$

Where $M^`$ and $\grave{\grave{M}}$ are the real and imaginary parts of the electrical modulus, and $\grave{\mathcal{E}}$ the real and $\grave{\grave{\mathcal{E}}}$ the imaginary permittivity.

The two equations (7) and (8) were used to calculate the electric modulus. The change in the real part ($M^`$) of the electric modulus versus frequency is shown in Figure-11 for 8 wt. % of PEO/alum films doped with carbon black at temperature (T=40 $^o$C). It can be clearly seen that values of ($M^`$) increase with frequency and reach a rather constant value. Finally, increasing the content of PEO/alum composites decreases the values of ($M^`$) as shown in Figure-12.

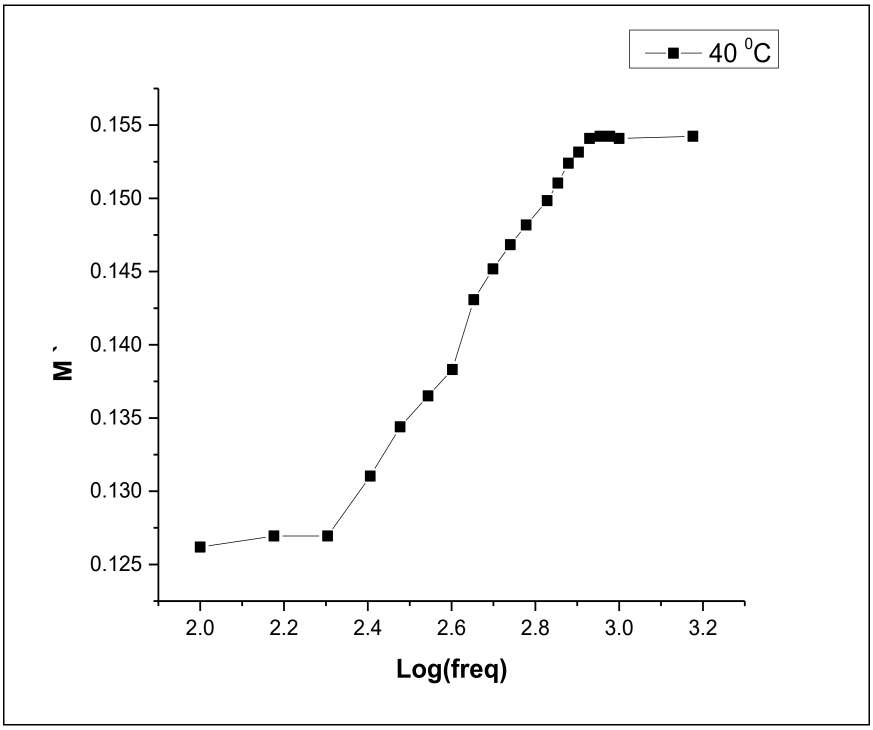

Figure-11. Electric modulus versus Log frequency for 8wt.% PEO/alum.

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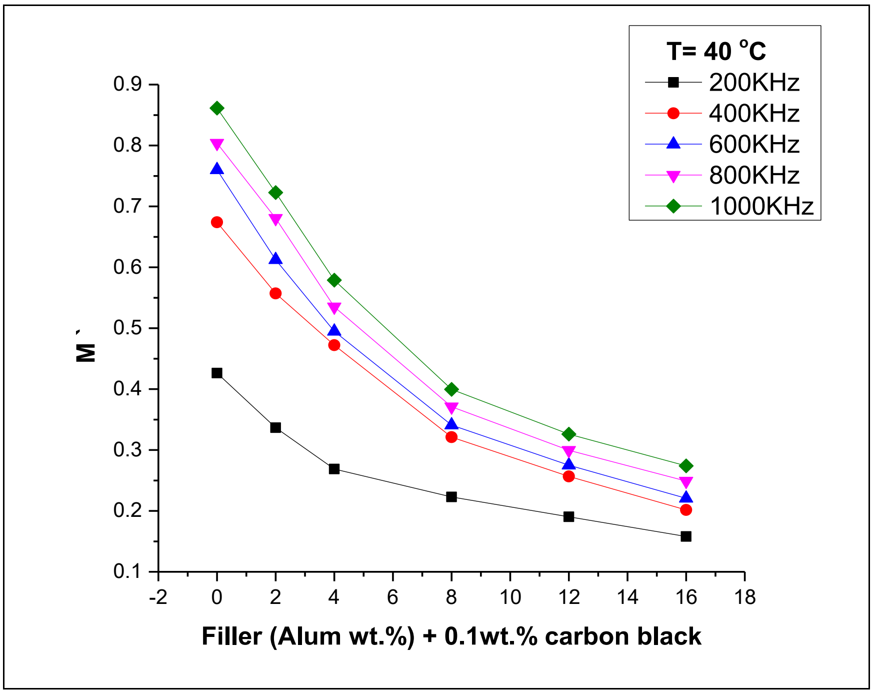

Figure-12. Variation of electric modulus with alum concentrations.

The change in imaginary part ($M^{``}$) of the electric modulus with frequency is shown in Figure-13 for 8 wt.% of PEO/alum thin films doped with carbon black at temperature (T=40 °C). The $M^{``}$ peak appears with temperature 40 °C, which indicating ionic conductivity a relaxation process. Finally, an increase in the content of PEO/alum composites increases the peak values of ($M^{``}$). And in the electric modulus presentation, the peak are formed their maxima increase with concentration of PEO/alum at $M^{``}$= (0.00064) and they shift slightly at the same time towards the left side of the frequency spectrum, thus providing means for study of relaxation [37]. These processes of relaxation were affected by the effect of the interfacial polarization that create electric charge collection around particles of alum and the peak position shift to higher frequencies refers to the relaxation processes related with (PEO) [38].

The position of the peak in ($M^{``}$) indicates the range in which the ions drift to long distances. In the frequency range which is above that of the peak, the ions are locally limited to potential wells and free to move within the wells. The frequency range, where the peak takes place, is suggestive of the transport from long range to short-range mobility [39]. Finally, an increase in the content of PEO/alum composites decreases the values of ($M^{``}$) as shown in Figure-14.

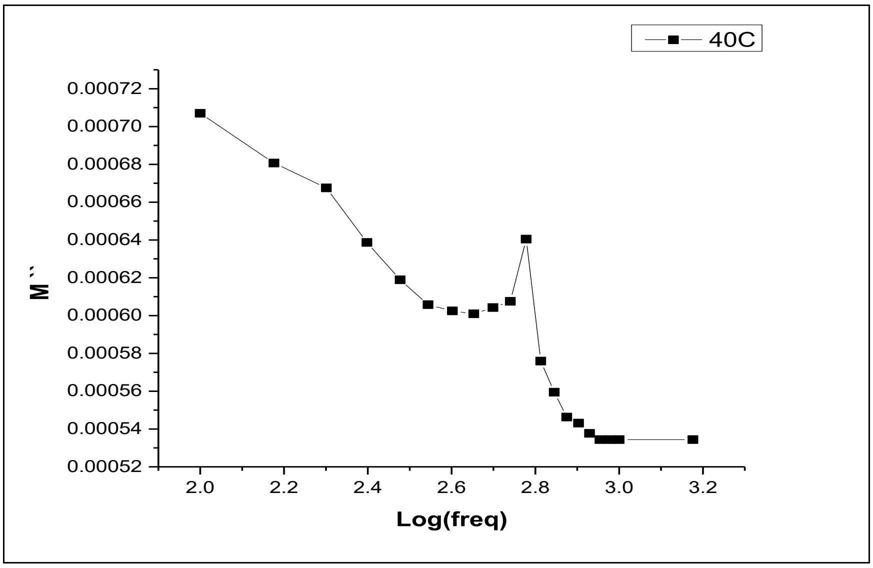

Figure-13. Imaginary part of electric modulus versus frequency for 8 wt.% PEO/alum concentrations.

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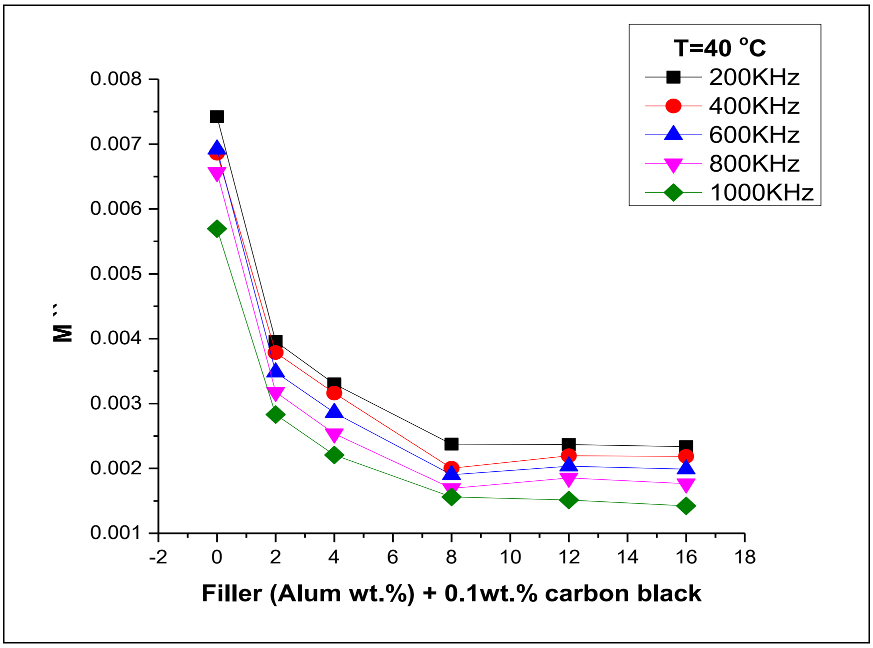

Figure-14. Variation of Imaginary part of electric modulus with alum concentrations.

4. CONCLUSIONS

The electrical properties of PEO/alum electrolytic thin films with different alum concentration and CB dopant at temperature (T=40 °C) were studied. By analyzing the results obtained, it was found that the impedance decrease with increasing frequency and filler concentration. While the dielectric constant and the dielectric loss of the composites increase with alum concentration and decrease with frequency. The AC conductivity and electric modulus vary with the applied frequency and alum concentration. The AC conductivity increases with increasing alum concentration and frequency. The electric modulus exhibit a relaxation peak at frequency 500 kHz. Fitting the observed data with some proposed empirical physical laws seems to be reasonable.

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