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Effect of high temperature on the Electric Properties of PEO/Potassium aluminum sulfate Composite Dopant with CB Nanoparticles

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Abstract

Electrical properties of the prepared polymer films, made of poly(ethylene oxide)(PEO) filled with electrolyte potassium aluminum sulfate 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=55 ^{0}C. The observed physical constants of the casted thin films like AC conductivity, phase angle, impedance, and dielectric constant were determined. It was found that these measured quantities vary with the potassium aluminum sulfate content and applied field frequency. The AC conductivity (σ_{ac}) increases with increasing potassium aluminum sulfate concentration and frequency. The dielectric constant (ε') and the dielectric loss (ε'') of the composites decrease with frequency and increase with potassium aluminum sulfate concentration.

Keywords: PEO, CB, AC conductivity, dielectric, phase angle, impedance.

1. INTRODUCTION

Materials with conductive properties are very important in everyday life. For instance, the transport of electrical energy from the power source to the light bulb, a good conductor is used, so that the electrical losses are kept minimal. Metal have the best conductive properties known in nature, but also semiconductors such as silicon, which is applied in integrated circuits, can transport current (electrical energy). Since the 1970's, a lot of attention has been given to the development of nonmetallic materials

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that can transmit electricity, such as conducting polymers and conducting composite materials [1].

Materials such as glass, ceramics, polymers, and biocomposites are non conducting materials. They prevent flow of current through them. When these types of non-conducting materials are placed in an electric field, they modify the electric field and they themselves undergo appreciable changes as a result of which they act as stores of electrical charges. When charge storage is the main function, the materials are called dielectrics. For a material to be a good dielectric, it must be an insulator. As good insulators, polymers posses excellent dielectric properties. Many authors have reported theoretical and experimental work related to these properties [2-3].

PEO has good chemical stability, both acid and alkali - resistant, corrosion-resistant. It is useful to enhance water dispersibility and water-based coatings, anti dusting agent in agricultural formulations, coupling agent, humectants, solvent, and lubricant in cosmetics and personal care bases, dye carrier in paints and inks, Low volatile, water soluble, and noncorrosive lubricant without staining residue in food and package process, plasticizer to increase lubricity, softener and antistatic agent for textiles [4].

The potassium aluminum sulfate filler 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 potassium aluminum sulfate with sulfates of singly cations of potassium and other elements. 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
andcomposedcharge transfer complexes(CTC),or may exist in the form of molecular combination between the polymer chains [5]. Carbon black 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 carbon black used as filler in polymers to import thermal and electrical conductions, i.e., conductive polymer composites [6-9].

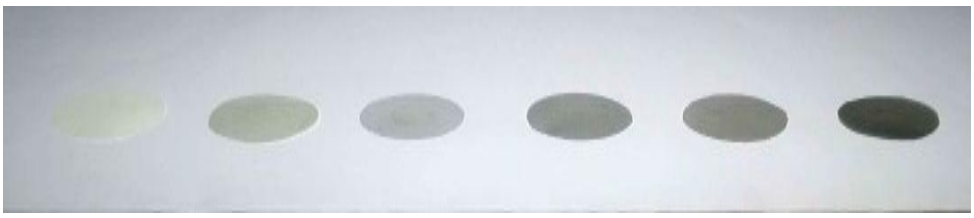
The electrical characterization is an essential for the industrial development of thin films of new polymers, composites and advanced materials that can be used as optical devices, polarizers filters, total reflectors, and narrow pass-band filters [10].

In the present comprehensive paper the studied PEO thin films are doped with carbon black nanoparticles at T=55 ^{o}C. Also, this paper covers electrical conductivity measurements and temperature effect T=55 ^{o}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=55 ºC. 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 potassium aluminum sulfate was ground into a fine powder of average particle size 18 µm. The powder kept in an oven overnight at 40 ºC to decrease the water content. The electrolyte potassium aluminum sulfate filler has a chemical formula **(**$KAl(SO_4)_2 . 12 H_2O$**),** 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/potassium aluminum sulfate dopant with carbon black nanoparticles by casting from solution made of Poly(ethylene oxide) and potassium aluminum sulfate . Poly (ethylene oxide) powder, potassium aluminum sulfate 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 potassium aluminum sulfate 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/potassium aluminum sulfate composites doped with CB

2.2. Electrical Measurements

The AC electric properties of the PEO/Potassium aluminum sulfate composite dopant with carbon black were studied through measurements of the impedance (Z), and the phase shift angle (φ) by using 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 (55 ºC). 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.

3. RESULTS AND DISCUSSION

The potassium aluminum sulfate 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/potassium aluminum sulfate 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/potassium aluminum sulfate 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 potassium aluminum sulfate and pure (polyethylene oxide) at (T=55 ^oC). 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 potassium aluminum sulfate 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=55 ^oC). 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.

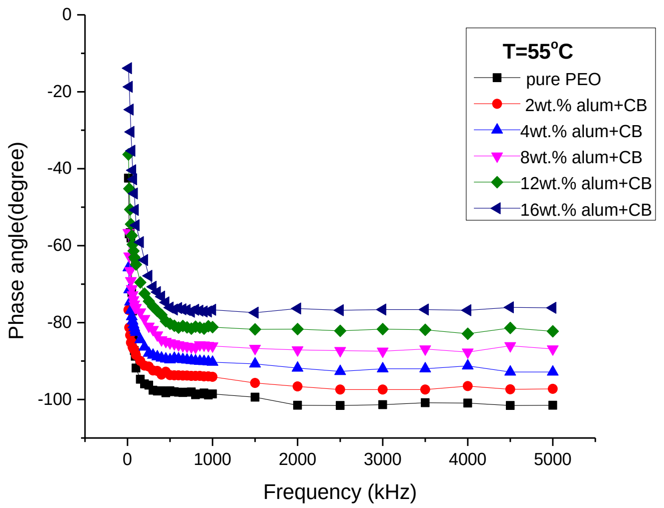


Figure-2. Phase angle versus frequency for PEO/potassium aluminum sulfate samples

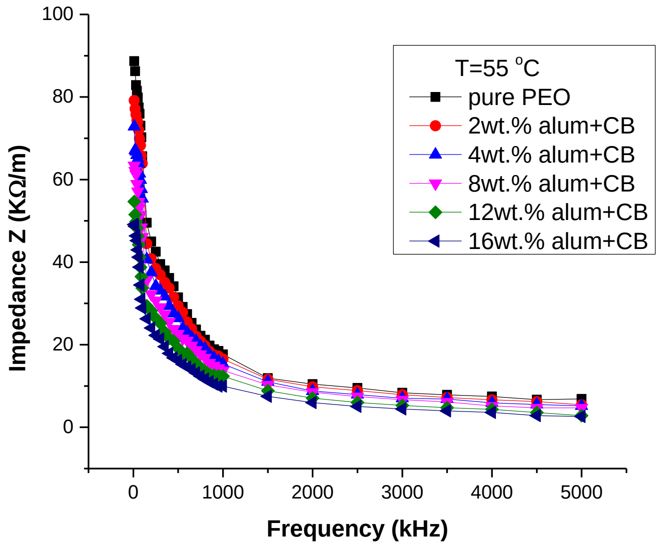


Figure-3. The variation of impedance with frequency for PEO/potassium aluminum sulfate samples

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 Poly(ethylene oxide) is lower than that of composite samples. The noticed progress in the Poly(ethylene oxide) insulation property is referred to electrons and charge complexes incorporated in the potassium aluminum sulfate bulk and the conductive carbon black dopants, respectively, as seen from increasing the potassium aluminum sulfate concentration with the increase of the dielectric constant.

Figure-5 displays the behavior of the dielectric loss (ε") at (T=55 ºC), 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 potassium aluminum sulfate , and the charge carriers in carbon black are electrons, which cause large losses at lower frequencies, and to increase polarization influence [18].

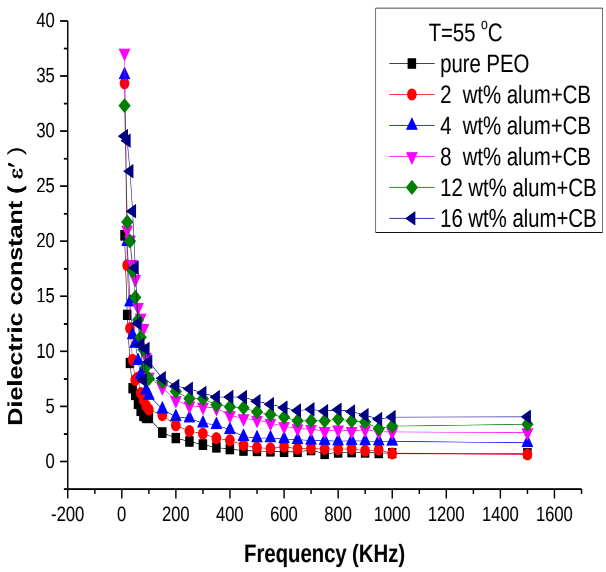


Figure-4. Dielectric constant versus frequency for potassium aluminum sulfate /PEO composite

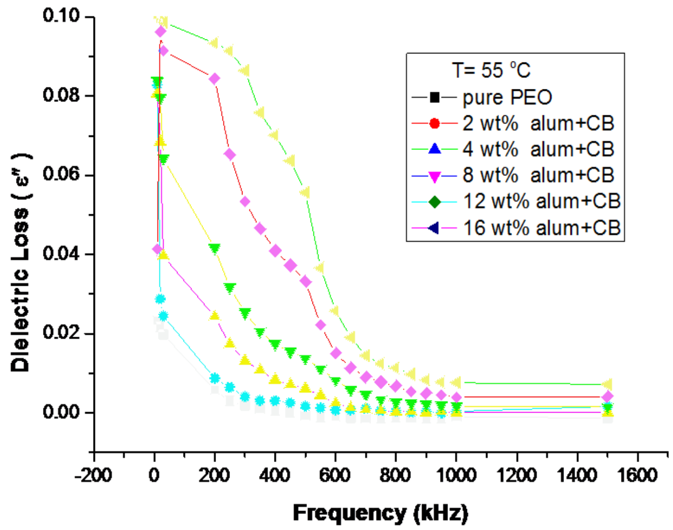


Figure-5. Dielectric loss versus frequency for potassium aluminum sulfate /PEO composite

From (ε') and (ε"), specifically at low frequency, a frequency dependence is observed, which represents the behavior of the polar materials as potassium aluminum sulfate [19]. The polarity of potassium aluminum sulfate is due to difference in electronegativity between aluminum and potassium, and the geometry of potassium aluminum sulfate 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 potassium aluminum sulfate and Poly(ethylene oxide) polymer of the polar semicrystalline polymers i.e., dipole rotation or polar polarization, the PEO/potassium aluminum sulfate and dopant carbon black composites are effectively operating under the high electric field.

Figure-6 shows that with increasing the frequency and potassium aluminum sulfate concentration, the AC conductivity (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 potassium aluminum sulfate 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, specially 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].

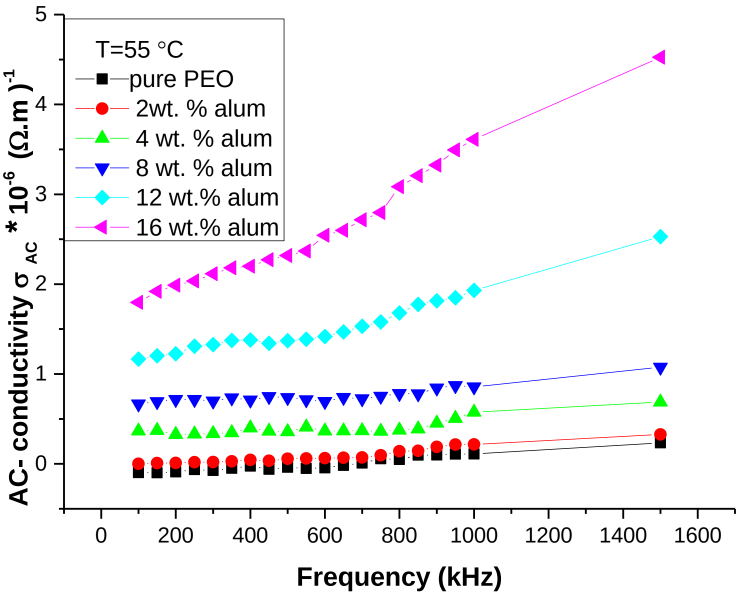


Figure-6. Dependence of AC conductivity as a function of frequency

3.2 The Relation Between Potassium aluminum sulfate Concentration and Electrical Properties

Figure-7 shows that, at different frequencies, impedance decreases when the potassium aluminum sulfate filler substance increases at temperature 55 ^{o}C. This decreasing in (Z) refers to free electrons and ions transfer, due to impurities in the potassium aluminum sulfate and carbon black dopant. The electrical conduction increases because of these reasons.

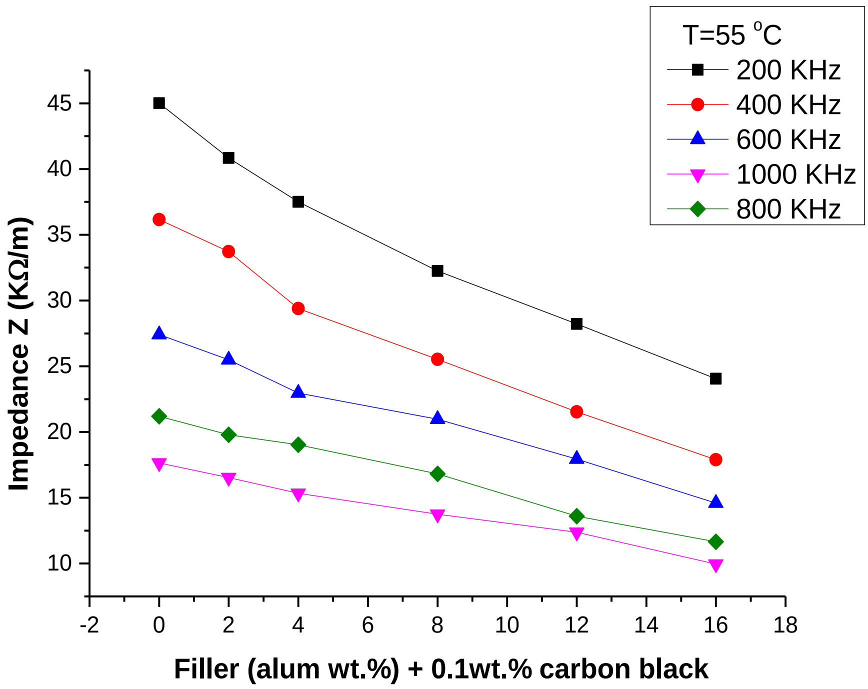


Figure-7. Variation of impedance with potassium aluminum sulfate concentrations

Figure-8 displays that with increasing the frequency, the dielectric constant (ε') decreases, while it increases greatly from about 0.56 for neat polyethylene oxide to 6.84, with increasing the potassium aluminum sulfate concentration up to 16wt.% . The high increment in (ε') with concentration of potassium aluminum sulfate 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].

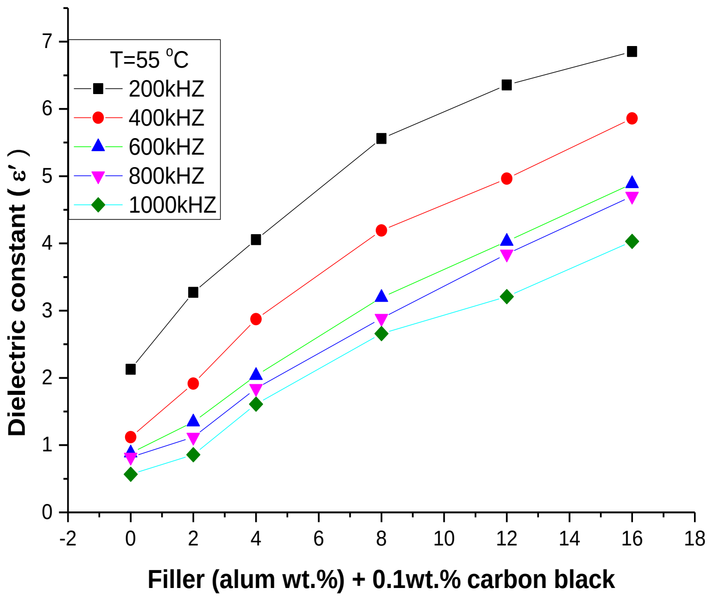


Figure-8. The dielectric constant versus potassium aluminum sulfate concentrations

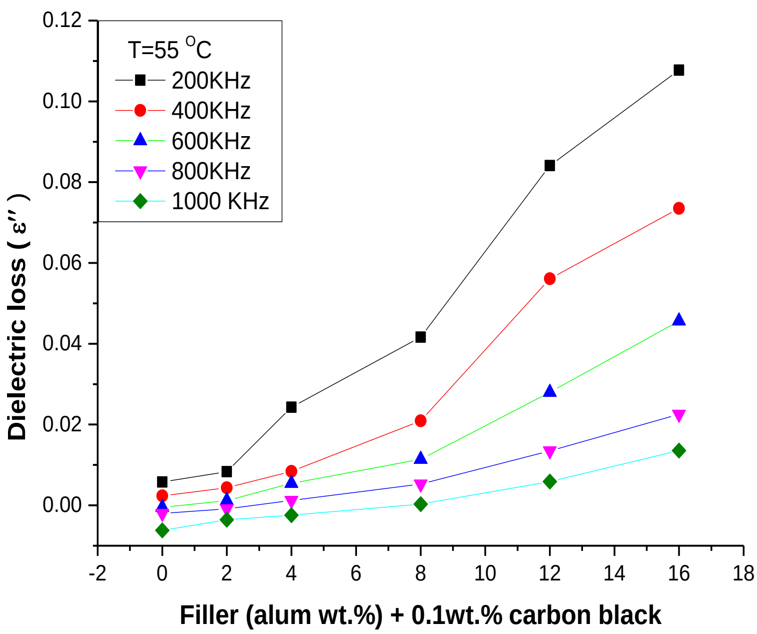


Figure-9. The dielectric loss versus potassium aluminum sulfate concentrations

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 oxide/potassium aluminum sulfate composites decreases. The increase in ionic conductivity with the potassium aluminum sulfate 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 Poly(ethylene oxide) 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 carbon black nanoparticles 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 Poly(ethylene oxide) 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.

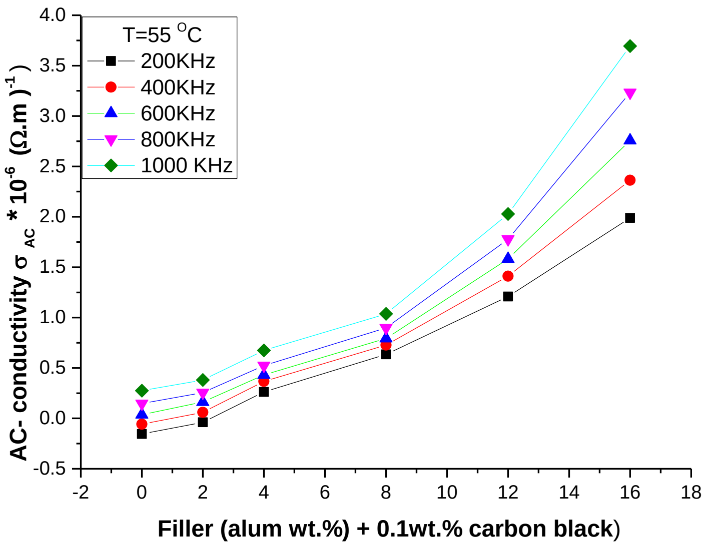


Figure-10. AC conductivity versus potassium aluminum sulfate concentrations

It was found that AC-conductivity values increase Under the effect of temperature (55 ºC) at all frequencies, and this is due to electron and impurity activation that increases with temperature, and to ionic and molecular mobility of Poly(ethylene oxide) 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 Poly(ethylene oxide) 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 potassium aluminum sulfate 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 the movement of carbon black nanoparticles in the thin films bulk that are trying to create paths network [34-35].

4. CONCLUSION

The electrical properties of PEO/potassium aluminum sulfate electrolytic thin films with different potassium aluminum sulfate concentration and carbon black nanoparticles dopant at temperature (T=55 ºC) 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 potassium aluminum sulfate concentration and decrease with frequency. The AC conductivity and electric modulus vary with the applied frequency and potassium aluminum sulfate concentration. The AC conductivity increases with increasing potassium aluminum sulfate concentration and frequency. Fitting the observed data with some proposed empirical physical laws seems to be reasonable.

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