Effect of Potassium alum and Carbon Black nanoparticles on casted PEO composite: phase angle, dielectric constant, impedance and AC conductivity

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Abstract—The electrical characteristics of hybrid polymer thin films consisting of conductive carbon black (CB) nanoparticles (0.1wt%) doped in poly(ethylene oxide) (PEO) filled with electrolyte potassium alum salt at varied concentrations were studied. For varied potassium alum concentrations and fixed content of conductive carbon black of concentration (0.1 wt. percent), the AC electrical characteristics were studied in the frequency range (3kHz - 5MHz) and temperature range (30 ºC - 55 ºC). Thin film physical constants such as dielectric constant, dielectric loss, AC conductivity, and impedance have been recorded. These measured amounts were discovered to fluctuate with potassium alum concentration, applied field frequency, and temperature. With increasing potassium alum content, frequency, and temperature, the AC conductivity (ac) increases. Dielectric constant (ε') and dielectric loss (ε") of the composites increase with potassium alum concentration and decrease with frequency.

Keywords: PEO, CB, potassium alum, AC conductivity, dielectric constant, dielectric loss.

I. INTRODUCTION

Normal Solid polymer electrolytes (SPE) are materials that are yet promising for use in electrochemical and other devices. Solid batteries, transistors, smart sensors, and other solid-state devices are among the applications for these SPE films. They are constructed from a polymer distributed in an electrolytic ionic material to generate ionic systems in which electricity is transferred mostly by ions diffusion under the applied electrical field. They're now employed in high-energy-density batteries that can be recharged, full-cell integrated optical systems, and electrochromic displays with great success. Polyacrylonitrile and polacrylamide are two polymers that are employed as basic materials in the production of polymeric electrolytes thin films [1-5].

Poly(ethylene oxide) has good thermal and electrical insulation properties, it is white solid with melting point around (65 ºC) low toxicity, Low glass transition temperature (-65 ºC) enables for ion transport at ambient temperature. Because of its large ability to dissolve with ions, superior processability, and distinct mechanical properties, (PEO) is the best polymer employed, because its large ability to dissolve with ions, good process ability and distinguished mechanical properties [6-8].

The potassium alum filler comes in the form of transparent glassy crystals that are water insoluble. It is a solid electrolytic ionic salt formed by hydration of a single cation sulfate (K^{+1}) and a triply charged sulfate (Al^{+3}) to generate potassium alum with sulfates of singly cations of potassium and other elements. Water purification, dyeing, fireproof fabrics, and leather tanning are all frequent applications. The addition of electrolytic elements has a stronger impact on the composite's bulk properties and ionic conductivity. The interactions between a polymer matrix and an alkaline ion influence its potential applicability in solid-state electrochemical devices and energy batteries. In the composite bulk, the ion conduction was attributed to impurity activity and ionic interaction occur the passage of a current into the sample.

When carbon black (CB) is doped in solid polymers, it can exist in the form of molecular aggregates between the polymer chains or it can go substitutionally into the polymer chains and create charge transfer complexes [9]. Carbon black is a non-

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crystalline shape that is conductive and made up primarily of atoms for carbon or aggregates with a spherical shape. The primary use of (CB) as a polymers filler is to introduce electrical and thermal conductivity, resulting in conductive polymer composites [10-13].

The thermal, electrical, mechanical, and optical characterization of thin films of new advanced materials, polymers, and composites that can be employed as polarizers, filters, optical devices, and narrow pass-band filters total reflectors, is critical for their industrial development [14]. The investigated PEO/potassium alum films are doped with (CB) nanoparticles in this research, resulting in hybrid films. This work also discusses AC electrical conductivity measurements and the effect of temperature on a few physical parameters, focusing on a more detailed characterization and a better understanding of electrical conductivity in doped films. The AC electrical conductivity is investigated as a function of frequency in the range (3 kHz - 5 MHz) and different temperatures (30 ºC -55 ºC). Quantities of measured as impedance, dielectric constant, dielectric loss, and AC conductivity were calculated.

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II. Materials and Methods

A. Materials and composites thin films preparation

Potassium alum (potassium sulphate) came in the form of crystals and was water insoluble. To reduce the water content in samples, solid alum was ground into a powder with an average particle size of about 20 µm and stored in an oven at 40 oC. Potassium alum is an electrolyte made up of a sequence of crystallized double sulfates with a tiny quantity of impurities. Its chemical formula is (KAl (SO4)2. 12 H2O). By casting composite films of PEO/potassium alum dopant with carbon black from a solution of PEO and potassium alum, poly (ethylene oxide) with an average molecular weight of 300,000 g/mol was utilized. The powders of poly(ethylene oxide), potassium alum, and carbon black were mixed together.The mixture was then swirled continuously at room temperature for three days with a rotating magnet until it had an uniform viscous molten look. The liquid was poured into thin sheets on a glass mold right away (plate). Waiting two days under air pressure allowed the methanol to entirely evaporate at ambient temperature. For two days, all of the samples were dried in a 40oC oven. A vernier caliper (digital) is used to measure the thickness of the composites. The vernier caliper's smallest division is 0.001 mm. All thin film thicknesses are measured at a number of different locations, chosen at random, and the average is used in the count. The obtained average thickness value is around 100 µm. Figure 1 shows the situation.

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

B. Electrical measurements

The AC electric properties of the PEO/potassium alum composite dopant with CB were investigated using a Hewlett Packard (HP) 4192A impedance analyzer to evaluate the impedance (Z) and phase shift angle (φ).The values of impedance (Z) in ohm's (Ω) and the phase angle (φ in degrees) were measured directly using impedance analyzer. In a sample holder, the test specimens were firmly held between two copper electrodes. The impedance analyzer was connected to these electrodes via cables. Impedance measurements were taken across a temperature range of 30 oC to 55 oC in a frequency range of around 100 kHz to 3 MHz. The sample holder was put inside an oven chamber to evaluate the influence of temperature on impedance and phase angle. Temperature readings were taken in a precise manner. In addition to the temperature readings on the oven dial, a thermocouple implanted beside the test specimen was used to collect accurate temperature data in a steady state condition. The temperatures were taken below the melting point of PEO, which is around 60 degrees Celsius.We have calculated the dielectric constant, AC conductivity and other values of PEO/potassium alum composite dopant with CB composites films. The calculations by using the analysis equations and graphical representations were done using computer and special software programs.

III. Results and Discussions

To compute the electrical characteristics of PEO/potassium alum composite with carbon black dopant in various cases, some theoretical relations are necessary, such as: effective frequency of electric field, temperature and concentration of filler.Dielectric materials are a distinguished type of materials that are insulators under almost all cases. They have the useful and interesting property that their molecules, electrons or ions, under the effect of an electric field (external) may be polarized. When these composite are put between charged plates as in capacitors, and therefore increase the capacity. [15]. Connecting a capacitor (C) to a resistor (R) in parallel, (Z) is the impedance, (Z'') is the imaginary component and (Z') is the real component of the impedance of the circuit.

Since: Z = Z' – i Z''. We can separate the real component of the impedance Z' and the imaginary component of the impedance Z'' via multiplication by (1 – i ω C R), the complex conjugate. Thus:

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

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

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

These equations are used to compute the dielectric constant (ε'), which is associated to the energy stored in the medium, and dielectric loss (ε"), which is correlated to the energy loss in the medium in the form of heat caused by an electric field [16].

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

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

Where f is the electric field frequency (AC) and Co is the capacitance without the thin film,
AC conductivity (σAC) of the film is given by the equation

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

The potassium alum filler attached to the PEO matrix to formed electrolyte films is under consideration to estimate the function of the filler in the operation of the ionic conduction under effect of applied electric field. The purpose of investigating electrical conduction in PEO polymer is explain the nature and kind of the charge motion in conductor[17].

A. The field frequency effect on the electrical properties

Impedance experiments on PEO/potassium alum thin films doped with carbon black nano particles are carried out across a range frequency range (30 kHz - 3MHz). Figure 2 shows the relationship between field frequency and phase angle (φ) (the phase difference between voltage and current) for films with varying potassium alum concentrations and pure PEO. When the phase angle is negative, the bulk material can be made up of capacitive and resistive connections.

Figure 2 shows that with increasing potassium alum concentration the change of (φ) across minimum negative values, referring that the capacity will be less than resistivity of the PEO/potassium alum thin films doped with CB due to increase of ionic and electronic.

Figure 3 shows the variation of impedance (Z) per unit thickness with frequency. This figure shows that the impedance decreases with increasing frequency. The high impedance quantities at low frequency, because of effect of space charge polarization, electrode polarization in the samples, and various structural defects (grain accumulations ,phase boundaries) [18-19]. The quick decrease quantities of impedance show a response of the bulk to applied alternating field. This attitude may be indicated to decreasing of the interfacial polarization effect, which may found at the electrode surfaces thin films [20-21]. Due to the polarization effects the frequency increasing with the impendence decreases, the enhancement of electronic mobility and formation of connected carbon black paths.

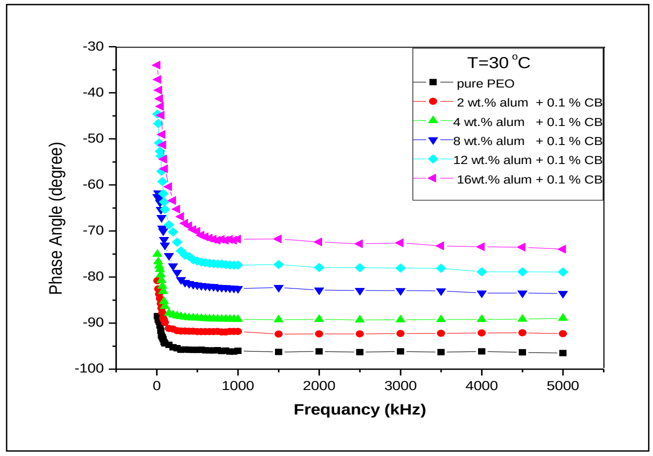

Figure 2. Phase angle versus frequency for PEO/potassium alum samples

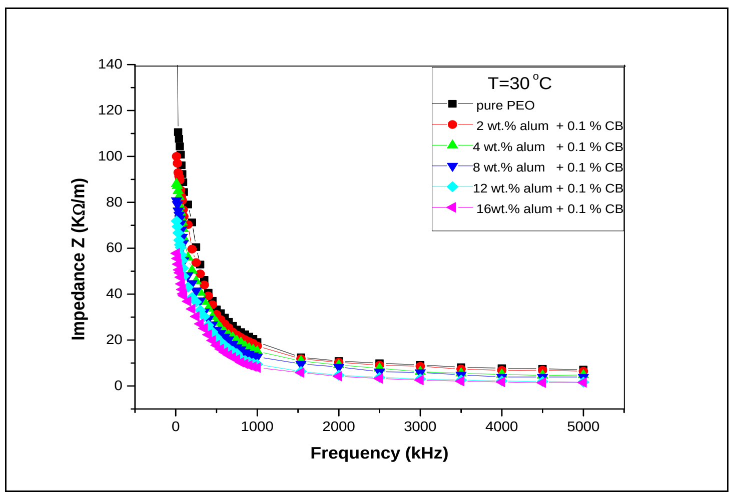

Figure 3. Impedance versus frequency for PEO/potassium alum samples

In figure 4, dielectric constant (ε') decrease with increasing frequency may be explain on the polarization of space charge which a little promotes and repairs for this behaviour take place by the orientation polarization. The dielectric constant (ε') of Poly (ethylene oxide) is less than that of composite thin films. The observed improve in the Poly(ethylene oxide) insulation property is indicated to charge complexes and electrons combined in the potassium alum bulk and CB dopants, respectively, Also deduced from increasing the concentration of potassium alum with increase of dielectric constant.

Figure 5 shows the attitude of the dielectric loss (ε"), at low frequencies dielectric loss has a high value, and then at a high level of frequencies it starts to decrease. The charge carriers in dielectric loss are expressed as electrons (carrying electrical charge) and ions (particles move freely). The ions (Al^{+3} and K^{+}) are charge carriers found in potassium alum, and the electrons are charge carriers in CB nono particles, causing high losses at low frequency and a high polarization effect [22].

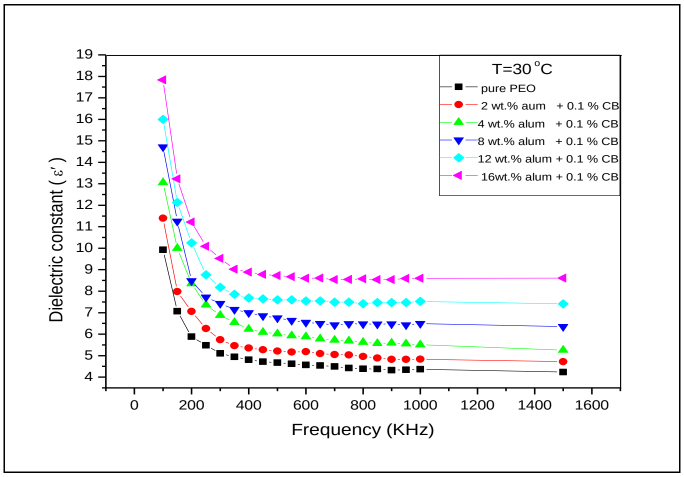

Figure 4. The variation of dielectric constant with frequency for PEO/potassium alum composite

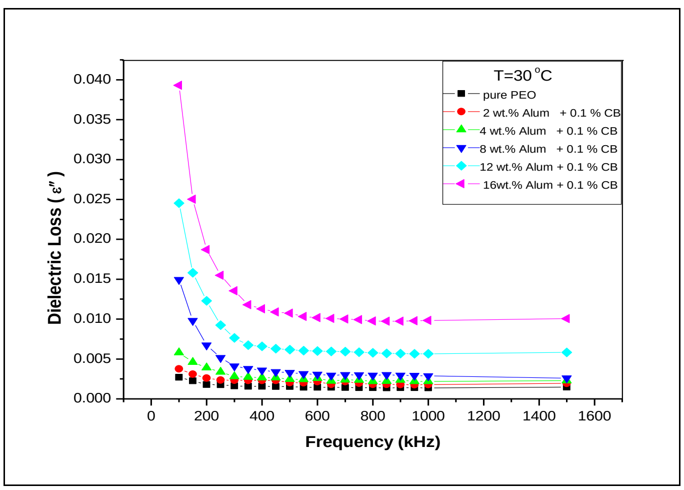

Figure 5. The variation of dielectric loss with frequency for PEO/potassium alum composite

From dielectric constant (ε') and dielectric loss (ε"), a frequency dependence is observed at low frequency, which represents the behavior of polar materials as potassium alum [23]. The polarity of potassium alum is attributed to electronegativity differences between aluminium and potassium, and the geometry of potassium alum is octahedral. It may readily be seen that with frequency of the electric field both (ε') and (ε") are decreased. These outcomes suggest of the potassium alum and PEO polymer of the polar semi crystalline polymers i.e., polar polarization or dipole rotation, the PEO/potassium alum and carbon black dopant thin films are efficaciously operating under high electrical field.

Figure 6 shows that as the frequency and potassium alum concentration increase, the AC conductivity (AC) values increase because more ions and charges may flow in a bigger electric field, causing the ionic conduction process to increase. This conclusion demonstrates that high frequency induces bulk (AC) conductivity [24-25]. Ionic conduction occurs when cation vacancies in ionic materials allow ionic transport in the direction of an applied electric field. It may be assumed that with higher frequencies and potassium alum content, ionic conduction is facilitated.

The electronic and ionic interactions in the bulk polymer electrolyte reason the increasing in the AC-conductivity and dielectric constants. An excess in movable charged particles and ions are created by bulk effect, principally the impurities [26].

CB nano particles compose continuous paths in the matrix of polymer. Through these paths move the free electrons under electric field. Depend on the well-known (conduction path theory), the motion of electrons causes the electrical conduction process [27]. AC conductivity increases with increased frequency and this is expecting as movable charge carries, resulting reinforcement mechanism of conduction. At high frequencies, the induced conductivity determines the level of semiconducting of the materials [28].

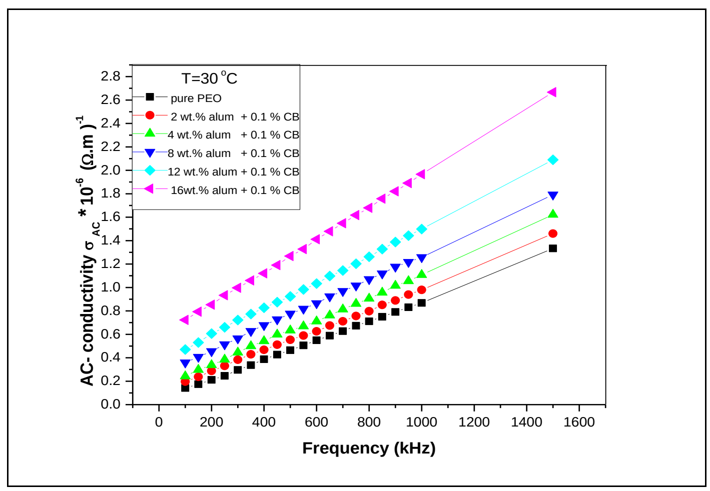

Figure 6. AC conductivity as a function of frequency

B. *The relation between potassium alum concentration and electrical properties*

Figure 7 illustrates that when the potassium alum filler substance is heated to 30°C, impedance reduces at various frequencies. This reduce in impedance indicate to ions and free electrons motion, due to potassium alum impurities and CB dopant. So that increases the electrical conduction.

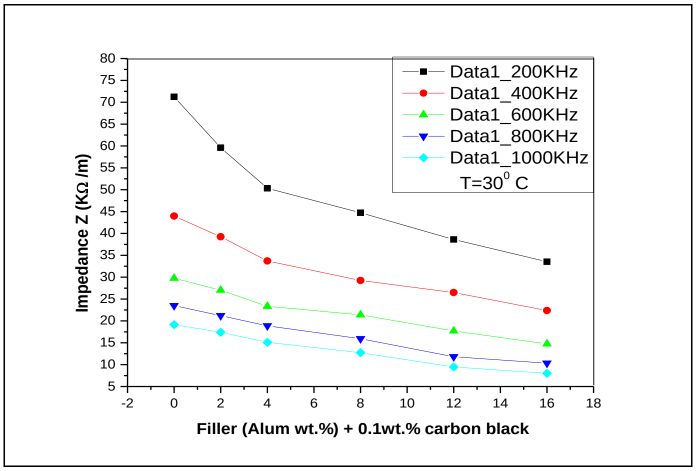

Figure 7. Variation of impedance with potassium alum concentrations

Figures 8 and 9 shows that with increasing frequency, the dielectric constant (ε') decreases, whilst it increases from about 4.3 for pure polyethylene oxide to 11.2, with increasing the potassium alum concentration up to 16wt.%. The high increase in dielectric constant with concentration of potassium alum is because of the contribution of ions (particles move freely) and free electrons (carrying electrical charge). Particles of carbon black (CB) make conductive paths which make the thin film extra conductive, resulting increase dielectric constant. Conduction behaviour of the thin films is controlled by the content of filler [29]. The increase of dielectric loss (ε") may expresses to the polarization (interfacial polarization) of such a heterogeneous system [30].

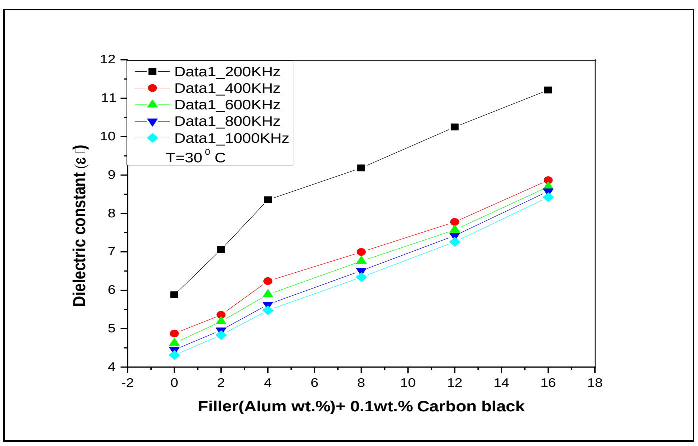

Figure 8. The dielectric constant versus potassium alum concentrations

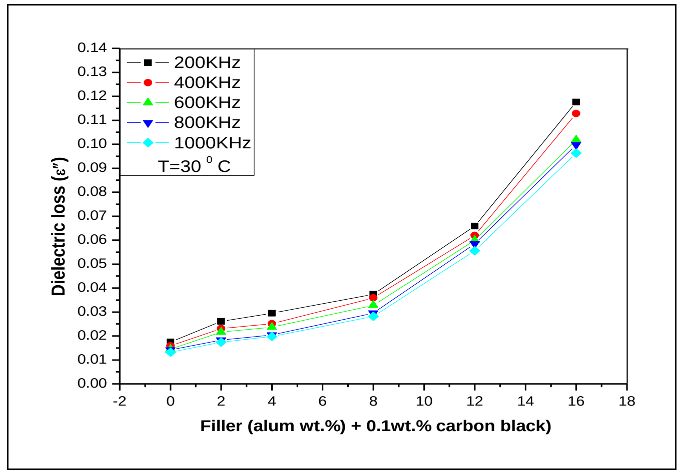

Figure 9. The dielectric loss versus potassium alum concentrations

Figure 10 shows that at various frequencies, with increasing potassium alum concentration the AC conductivity (σAC) increases, which means with increasing of the content filler the resistivity of PEO/potassium alum film decreases. The (AC) conductivity is roughly constant up to 1.7 wt.%, then it increases quickly with increasing the potassium alum concentration. Accordingly, the point of 1.7 wt.% filler content can be defined a critical point (a percolation threshold) for the samples. The increase of alum concentration is shown by an increase in ionic conductivity with the potassium alum content. The flow of ions through the polymer is corroborative by the great amplitude of the (PEO) segmental motion, whereas the movement of ions through the solid electrolyte is liquid. The crystalline area has a higher amorphous region than the segmental mobility of polymer chains. The continuous increase in polymer could be due to a decrease in crystallinity due to the casting process, or an increase in amorphism. Ionic size is one component that contributes to increased ion mobility, which leads to increased polymer conductivity. Because Al^{+3} and K^{+} are both fast conducting ions in various amorphous and crystalline materials, combining them in a polymer would improve its electrical and optical properties [31].

CB particles create large number of conductive paths within the polyethylene oxide matrix, which allows a current to move through the thin film. CB particles increase conductive paths, and it create a conductive network is [32].

The increase in AC conductivity (AC) due to carrying electrical charge (electrons) that successfully tunnel across isolated carbon black (CB) nano particle domains with reducing the spacing at low concentrations of carbon black [33]. The conductive channels become larger when the number of carbon black (CB) micro particles increases, and the distance between contiguous clusters decreases [34]. Because of the increased interaction between the CB nano particles, the value of conductivity increases, resulting in a more conductive network in the PEO composite as the CB content increases. CB particles also have a higher dielectric constant [35]. When carbon black nano particles are doped in a composite, the gap between the particles conductivity widens and narrows as the charges transport becomes more straightforward [36]. As a result of the creation of per electron conduction and collating paths, the AC conductivity increases.

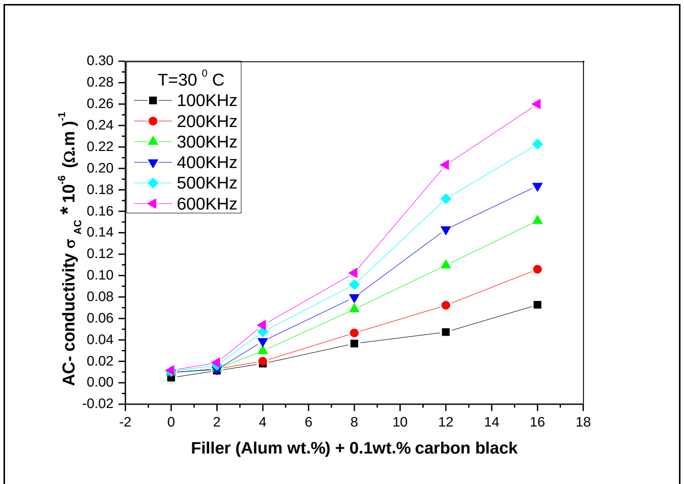

Figure 10. AC conductivity versus potassium alum concentrations

C. The relation between electrical properties and temperature

Figure 11 displays the variation of impedance (Z) with temperature at different concentrations of the potassium alum. It is obvious that increasing temperature decreases the impedance. This could be illustrated in specific cases like increasing charge mobility, generation of charge carriers, and reducing the energy gap. The charge carriers transport is affected by these processes in the thin film bulk, and these processes could occur from effects of conduction current and polarization superposition. The polarization currents are temperature dependent, where with increasing temperature the polarization decay time is reduced. This behavior illustrates that, as temperature increases the conductivity is increasing, such as the behavior of ionic and semiconducting materials [37]. While increasing the temperature impedance decreases due to the effects of polarization, forming of carbon black paths, and the increasing of movable electrons.

Figures 12 and 13 display the dielectric constant for 8 wt.% potassium alum filler and dielectric loss for 12 wt.% potassium alum filler versus temperature for different frequencies. With increasing temperature the dielectric constant (ε') increases. This is caused by a reduction in the polarization of space charge (interfacial) and formation of ionic and electronic arrangements or clusters in the polymer by heating. It is noticed that at high values of temperature (ε') will increase quicker. This illustrates that, the orientation of polyethylene oxide dipoles is simplified and relaxed when the temperature increases, so the dielectric constant increases [38].

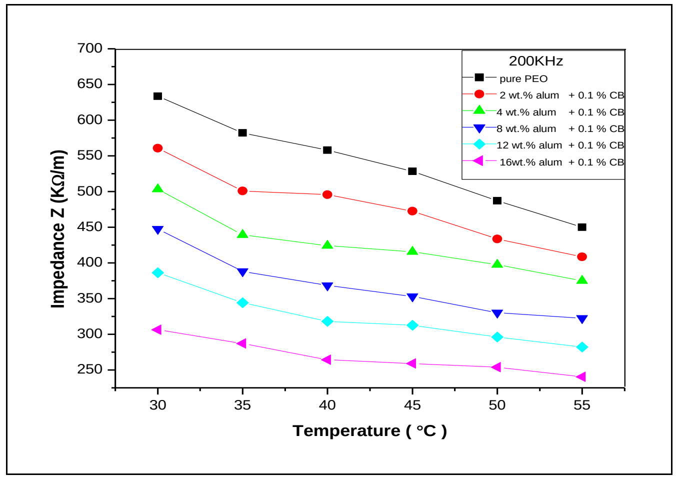

Figure 11. Impedance versus temperature for PEO/potassium alum composites

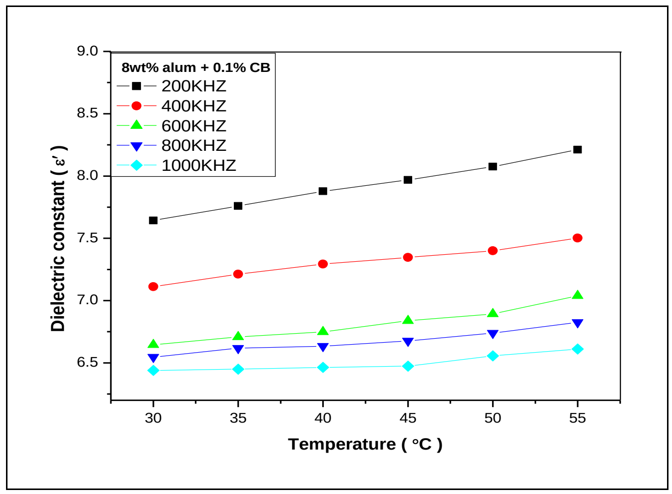

Figure 12. Variation of dielectric constant with Temperature

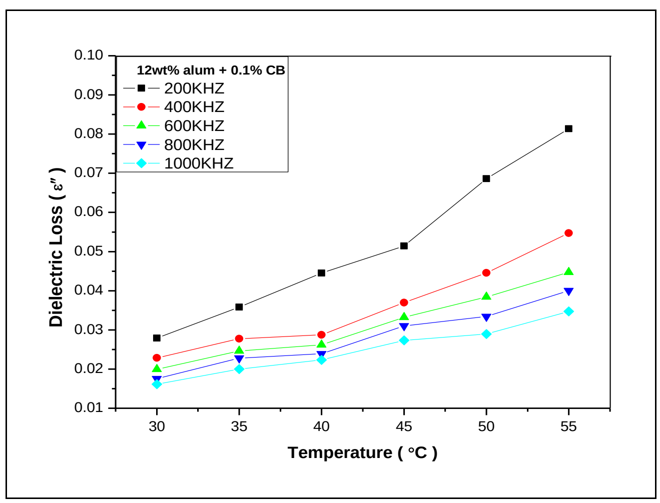

Figure 13. Variation of dielectric loss with temperature at different frequencies for 12 wt.% potassium alum

Figure 14 displays those at all frequencies; the values of AC-conductivity increase when the temperature increases. This is due to the activation of electron and impurity, which increases with increasing temperature, and because of molecular and ionic mobility of PEO stimulated when the temperatures increases, i.e., the electrons flow or charged ions found through the electrodes surface with rising motion chains of polymer and thus leading to high conduction.

The fast increase in AC-conductivity refers to the increasing of the charge carriers concentration as a consequence of increasing structure defects. Accordingly, new energy levels inside the forbidden energy gap can be generated; also, at high temperatures there is a stimulation of electronic mobility in impurities. In order to the temperature- conductivity behavior obey Arrhenius relationship, transport of cation is nearly like to that happening in ionic crystals, where ions jump into nearby vacant positions so that the ionic conductivity increase [39]. With increasing temperature, the movement of CB particles increase that are lead to Create percolation network [40]. At high temperature, Poly(ethylene oxide) matrix has sufficient mobility, thus CB nano Particles makes the conductivity of the composite increase [41].

As a function of frequency and temperature, the electrical conduction operation can be assembled as following:

- AC conduction indicates to impurities and charge of free electrons complexes in both potassium alum filler and carbon black dopant, the polymeric matrix.
- Maxwell-Wagner build-up polarization of interfacial charges at the composite interfaces.
- Hopping of energy levels and the free electrons over smaller barrier heights.

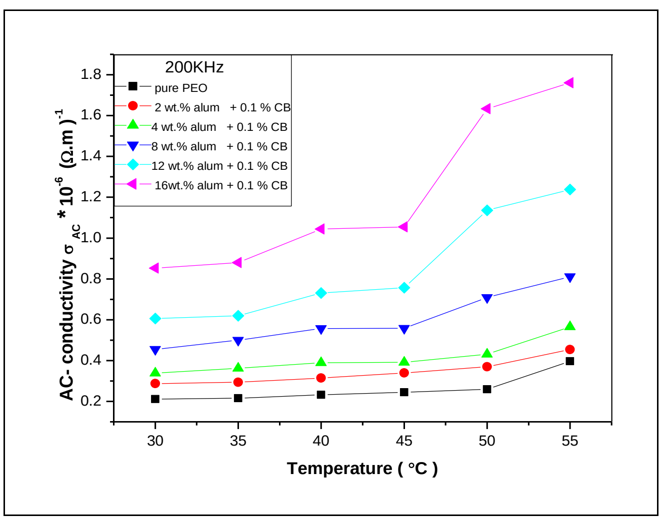

Figure 14. Variation of AC-conductivity with temperature for PEO/ potassium alum composites

IV. Conclusions

The AC electrical properties of Poly(ethylene oxide)/potassium alum films with different potassium alum concentration and carbon black dopant were studied. By analyzing the results obtained. The AC conductivity increases with increasing potassium alum concentration, frequency and temperature, which causes increases of process of ionic conduction. Impedance was found to reduce with increasing filler concentration, frequency and temperature due to the effects of polarization, forming of carbon black paths, and the increasing of movable ions and electrons. The dielectric constant and the dielectric loss of the thin films increase with both potassium alum temperature and concentration , and decrease with frequency due to the effects of polarization polar semi crystalline polymers i.e., polar polarization or dipole rotation. Fitting the data with some proposed empirical laws appear to be credible.

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