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ELECTRICAL MODULUS OF POLY (ETHYLENE OXIDE) COMPOSITES DOPED WITH CARBON BLACK NANOPARTICLES

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ABSTRACT

AC electrical properties were studied using the AC impedance technique. The electrical properties of thin films made of poly (ethylene oxide) (PEO) dispersed with do pants fixed amount of carbon black (0.1wt. %) were used in this study. The prepared films by casting method have been electrically. The present study has studying the change in the real part (*M* ') and imaginary (*M* ``) part of the electric modulus versus frequency of PEO thin films doped with carbon black at different temperatures, frequency in the range (200 kHz - 1000 kHz) and temperature in the range (30 ^{0}C-55 ^{0}C),and the study the real part (*M* ') part of the electric modulus for (PEO) with doped 0.1wt. % carbon black (CB) as compared to that case of the un doped (PEO) film. It was found that the real part (*M* ') of the electric modulus increases with frequency and decreases with temperatures with doped 0.1wt. % carbon black content. And it was found that the real part (*M* ') of the electric modulus decreases with doped 0.1wt. % carbon black content.

Keywords: poly (ethylene oxide) (PEO), carbon black (CB), electric modulus.

1. INTRODUCTION

Most polymeric materials are poor conductors (insulators) of electricity because of the unavailability of free electrons to participate in the conduction process. So a considerable interest has been focused on promoting their electrical conduction and developing their properties. Polymers display an assortment of electrical properties reflecting their molecular motion and structure. Electrical properties can be tailored to a specific requirement by the addition of suitable conducting elements or dopant materials [1-2].

Polyethylene oxide (PEO) is a familiar example of thermoplastic polymers which can be subdivided into crystalline and non-crystalline (amorphous) polymers. PEO has a property to form molecular complexes which enhance the electrical conductivity [3].Poly (ethylene oxide) (PEO) is a synthetic polyether that is readily available in arrange of molecular weights (Mw). PEO to polymers with a molecular weight above 200,000 g/mol, and PEO are liquids or low-melting point solids, depending on their molecular weights [4].

Carbon black is a conductive material and is largely composed of carbon atoms or aggregates of carbon atoms, although considerable quantities of hydrogen and oxygen can also be present. Carbon black are approximately spherical but the others have complex three-dimensional shapes, which can be thought of as the partial fusing together of a number of nanometer-sized spherical primary particles [5]. The most important CB use is as filler in polymers to improve electrical conduction and is used in preparation of conductive polymer composites.

2. EXPERIMENTALWORK

In this study the material examined is (poly ethylene oxide) thin films doped with carbon black as do pant and neat PEO sample for contrast the noticed consequences. The objective of the research is to investigate the AC electrical properties of casted Poly (ethylene oxide) thin films doped with conducting carbon black as compared to that case of the un doped (PEO) film.

2.1 Preparation of thin films

PEO and carbon black powders were blended together in methanol as a convenient solvent. Then for two days the mixture was mixed by using a rotary magnet to get a homogeneous mixture. On to a glass mould the mixture was directly casted to delicate films. At room temperature by waiting for two days the methanol was permitted to evaporate perfectly. All samples were dried in the oven at temperature 40ºC for two days.

The thickness of the synthesized composites was measured by a sensitive digital verniercaliper. Least count of the instrument is 0.001 mm. The thickness of thin films was measured at different spot places, chosen randomly, and then their average is taken. The thickness of (Pure PEO) film is 70µm and for (PEO/carbon black) film is 120µm.

2.2 Measurements of electrical properties

Hewlett Packard (HP) 4192A impedance analyzer was used to measure impedance and phase angle values by varying the applied frequency (ranges from 5Hz up to 13MHz), which is shown in Figure-1. The specimen was placed firmly between two copper electrodes in a sample holder shown in Figure-2. These electrodes are connected through cables to the impedance analyzer. Impedance measurements were performed in a frequency range from about 30 kHz up to 5 MHz over a temperature range (30ºC - 55 ºC) with steps of 5ºC. Since the melting temperature (T_m) for PEO is about 60 ºC, no higher temperature measurements were conducted. Temperature readings were taken in a steady state condition.

Figure-1. Hewlett Packard 4192A.

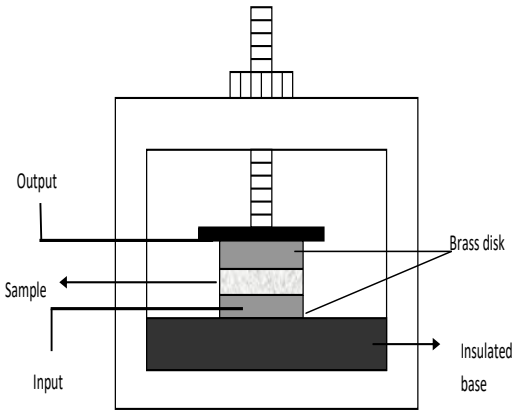

Figure-2. The sample holder diagramimpedance analyse.

Dielectric materials are a special class of substances that, under almost all conditions, are insulators. They have the interesting and useful property that their electrons, ions, or molecules may be polarized under the influence of an external electric field. When such materials are placed between charged plates as in capacitors, they increase the total capacity of these devices. This application constitutes one of the important applications of these materials [6].

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.

The complex impedance of the sample with the real and imaginary components can be calculated by:

$$Z' = Z \cos \varphi \quad (1)$$

$$Z'' = Z \sin \varphi \quad (2)$$

The dielectric constant ε' and the dielectric loss ε" of the sample are calculated from the following equations:

$$\varepsilon' = \frac{Z''}{2\pi f C_o Z^2} \quad (3)$$

$$\varepsilon'' = \frac{Z'}{2\pi f C_o Z^2} \quad (4)$$

Where f is the frequency of the applied ac electric field and C_0 is the capacitance of the two plates of the cell (capacitor) without the sample (dielectric).

Hence, a plot of Z' and Z" (or ε" and ε') yields a semicircle in the complex impedance diagram. The maximum value of Z' (or ε") is obtained when: $\omega_{max}\,\tau = 1$, which corresponds to the frequency: $\omega_{max} = 1/\tau$(where: $\omega_{max} = 2\pi f_{max}$).This equation enables the relaxation time to be calculated in a simple manner from experimental data. The graph of ε" versus ε' in the complex plane as an implicit function of frequency (called Cole-Cole plot) is shown in Figure-3.[7].

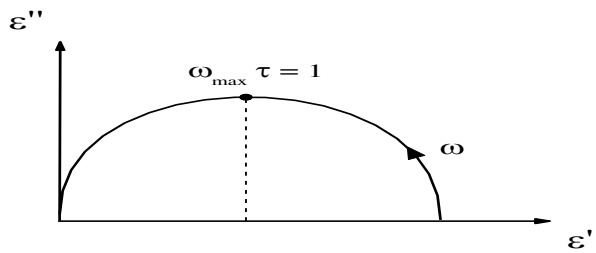

Figure-3. The Cole-Cole plot of ε" versus ε'.

To overcome this difficulty in the study of interfacial polarization, it has been decided to use the formalism "electric modulus", first introduced by McCrum *et al.* and Macedo *et al,* for the investigation of electrical relaxation phenomena in vitreous ionic conductors.

Electrical relaxation phenomena are usually analyzed in expression of the dielectric permittivity by the electric displacement vector (D) under the constraint of constant electric field (E). However, in dielectrics containing mobile charge, it seems convenient to concentrate on the electric field's relaxation under the constraint of a constant displacement vector, which leads to the inverse dielectric permittivity and the definition of electric modulus. An advantage 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 [8].

Complex modulus, electric modulus or inverse complex permittivity, M^*, is defined by the following equations:

$$M^* = \frac{1}{\varepsilon^*} = \frac{1}{\varepsilon` - j\varepsilon``} = \frac{\varepsilon`}{\varepsilon`^2 + \varepsilon``^2} + j\frac{\varepsilon``}{\varepsilon`^2 + \varepsilon``^2} \quad (5)$$

$$= M` + j\,M``, \quad j = (-1)^{1/2} \quad (6)$$

Where M` is the real and M`` the imaginary electric modulus, and ε` the real and ε`` the imaginary permittivity.

3. RESULTS AND DISCUSSIONS

The previous equations (1) and (2) were applied to calculate the electric modulus. The change in the real part (*M* ') of the electric modulus versus frequency is shown in Figure-4 for PEO/CB thin films doped with carbon black at different temperatures. It can be clearly seen that the values of (*M* ') increase with frequency and reach a rather constant value. An increase in temperature leads to a decrease in values of (*M* '). Finally, (*M* ') decrease with doped 0.1wt.% carbon black at temperature T=30^{0}C as shown in Figure-5.

The change in imaginary *(M* ``) part of the electric modulus versus frequency is shown in Figure-6. ForPEO thin films doped with carbon black at different temperatures. It can be clearly seen that peaks in the values of *(M``)* are developed, indicating ionic conductivity a relaxation process. Increasing the temperature shifts the peaks of (*M* ``) to higher frequencies. Finally PEO with doped 0.1wt.% carbon black composites increases the peak values of (*M* ``). And in the electric modulus presentation, peaks are formed their maxima increase with concentration of PEO/CB from (0.00047) to (0.00098) and they shift slightly at the same time towards the left side of the frequency spectrum, thus providing means for study of relaxation. 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) [9].

The (*M* ``) peak position 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 [10].

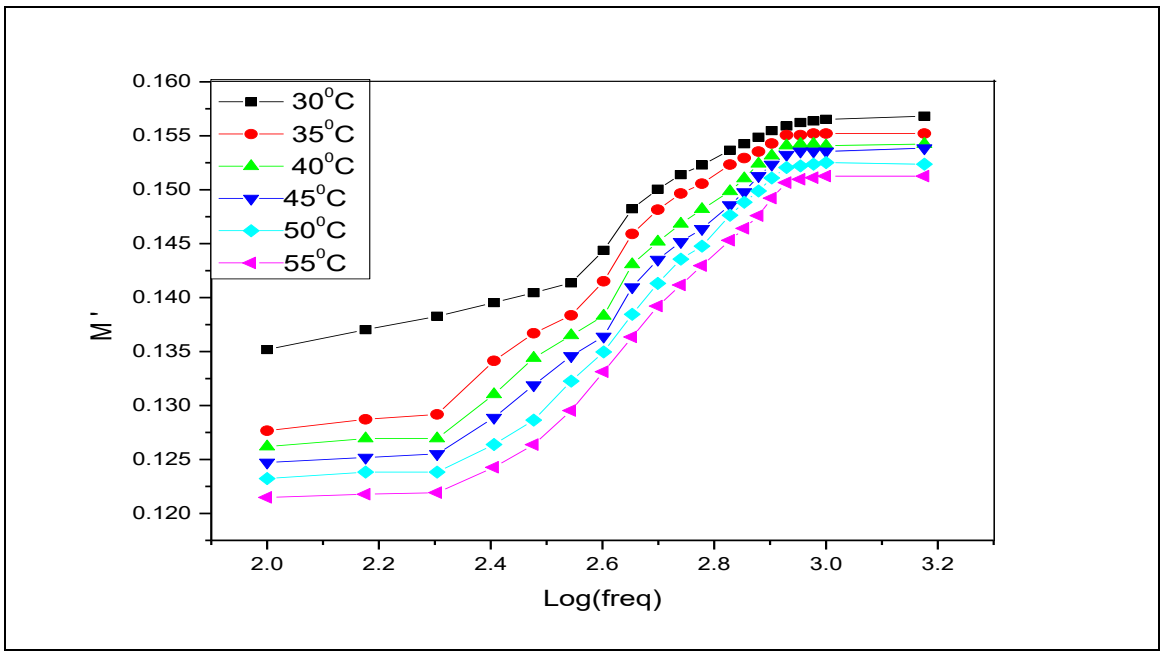

Figure-4. Electric modulus versus frequency for PEO/CB concentrations at different temperature.

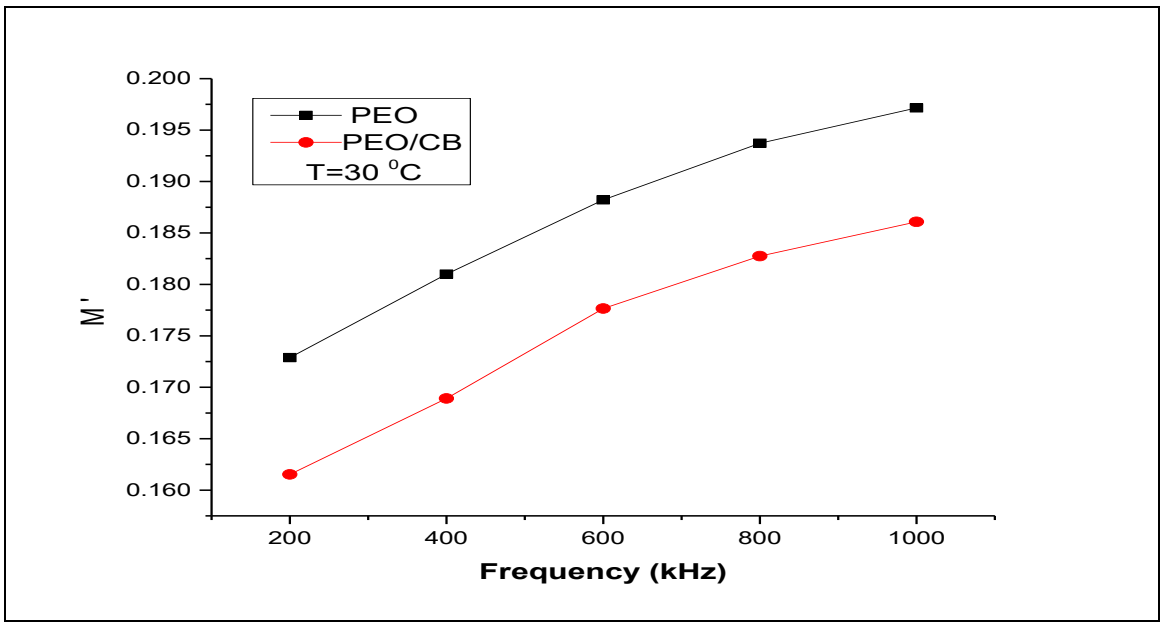

Figure-5. Variation of electric modulus with frequency.

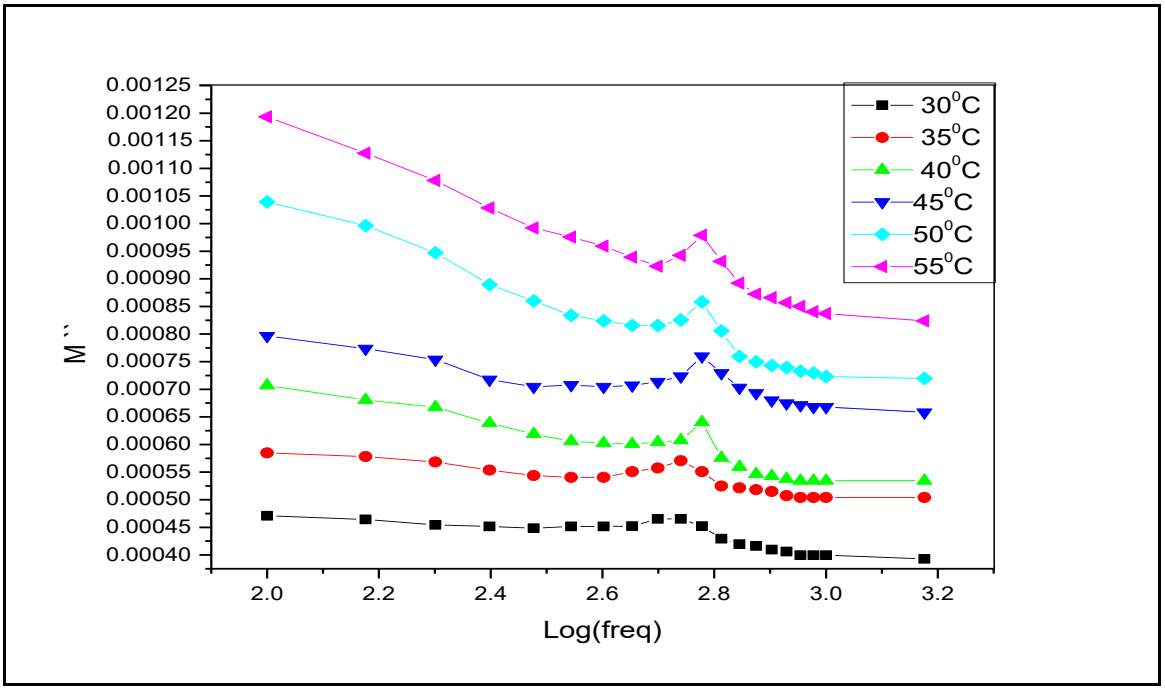

Figure-6. Imaginary part of electric modulus versus frequency PEO/CB concentrations at different temperature

Cole-Cole plots are usually used as a successful tool to analyze the dielectric data of dielectric materials. We use it here to characterize the dielectric behavior of the PEO/CB composites and find the relaxation time of these samples at different temperatures[11].

Figure-7 andFigure-8 show the Cole-Cole plots of the imaginary part of electric modulus versus the real part of electric modulus for PEO/CB at temperature (T= 30 °C and T= 55°C). And these Figures show the Cole-Cole plots the different composite sample of PEO/CB at different temperature, and the Cole-Cole plots of the imaginary component *M*`` versus the real component *M*` for each of the PEO/CB composites at different temperatures.

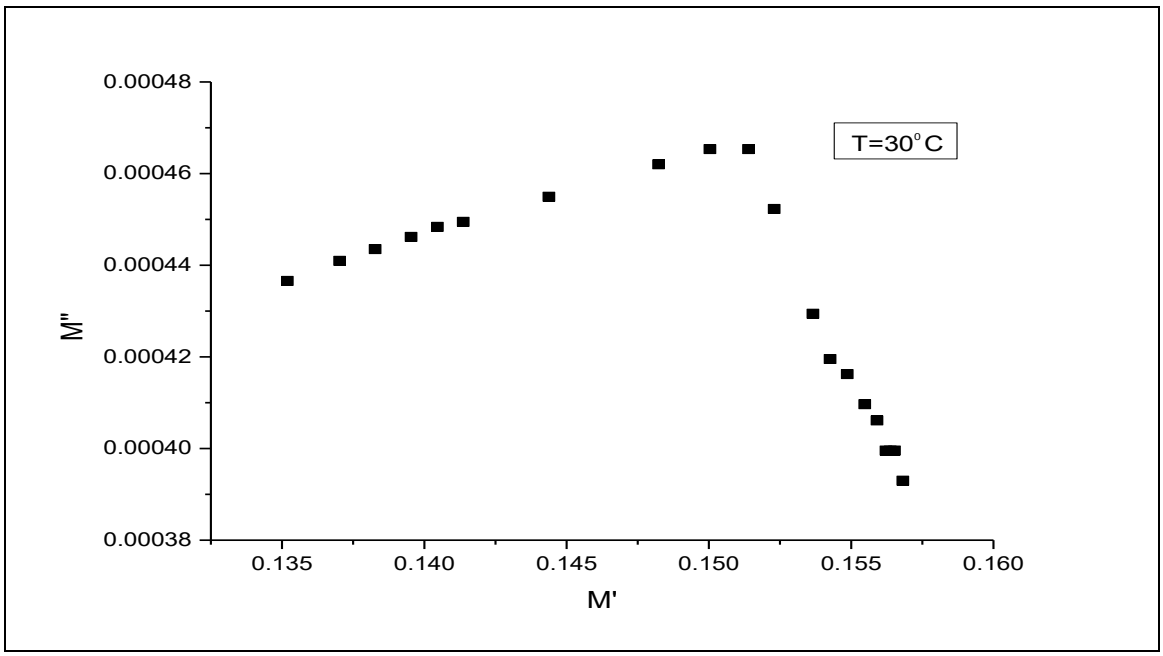

Figure-7. The Cole-Cole plots for of PEO/CB composite at T=30 °C.

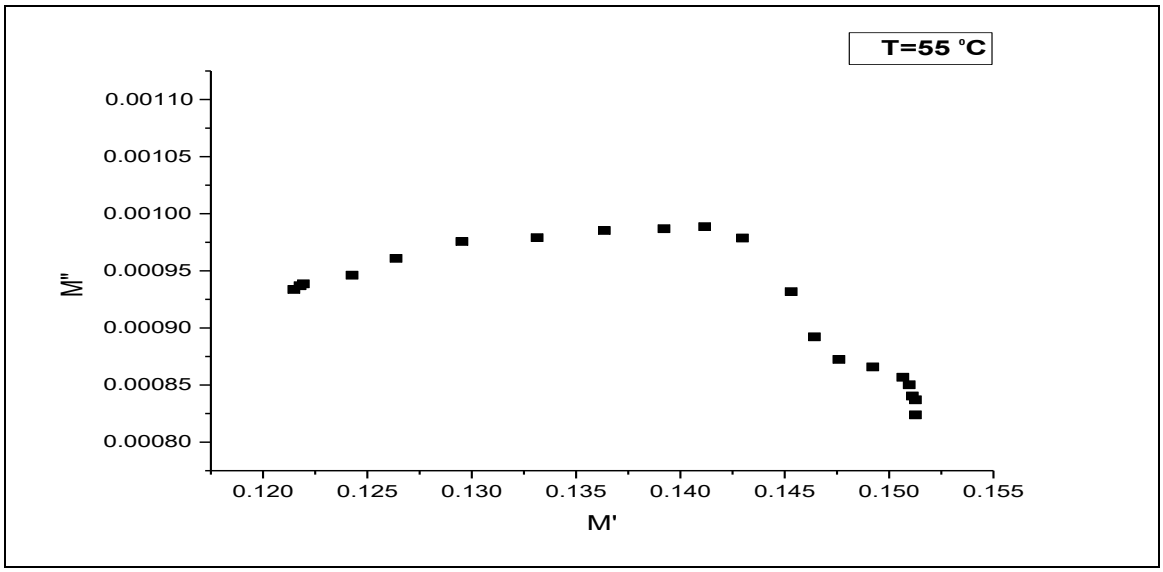

Figure-8. The Cole-Cole plots for of PEO/CB composite at T=55 °C.

The construction plane plots are distorted arcs exhibiting different electrical conduction processes with relaxation time spectrum.

The relaxation time (τ) is determined by approximating these Cole-Cole plots to semicircles using the relation:

$$\omega_{max}\,\tau = 1 \qquad (7)$$

where ω_{max} is the angular frequency at maximum values of ε'',*M*`` observed on the construction plots. The values of relaxation times of this specimen are inserted in table-1.

Table-1 shows that the calculated τ values decrease with increasing temperature; this is may be attributed to ionic mobility, which increases as temperature increase.

The relaxation time of the other two samples was calculated in the same way. The same behavior of the relaxation time was observed, since generally, the increase in temperature will increase the energy of the dipoles in sample so the dipole rapidly responds to the external electric field, i.e., increasing temperature decreases the relaxation time of the dipole rotation [12].

Table-1. Relaxation time for of PEO/CB composite as a function of temperature.

Temperature (^{o}C)	**Relaxation time** $\tau \times 10^{-6}$ (sec)
30	5.05
55	4.43

4. CONCLUSIONS

The electrical properties of poly ethylene oxide thin films doped with carbon black were studied. By studying the results, we deduced that:

a) The electric modulus increase with frequency and reach a rather constant value, and decrease with increase temperature, increase in dielectric constant.

b) The formalism of electric modulus is considered suitable for the investigation of the dielectric behavior of polymeric composite with a conductive component. It is capable revealing interfacial relaxation which, in most cases, is covered by the conductivity of the material.

c) The relaxation time showed variation with PEO/CB concentration temperature and frequency, and decrease with increase temperature.

d) Fitting the observed data to proposed empirical physical laws seems to be reasonable.

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