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## Research Article

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# **FTIR and dielectric relaxation analysis for PVC-Pb<sub>3</sub>O<sub>4</sub> polymer nanocomposites**

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## Abstract

This work studies the FTIR as well as dielectric characteristics of the PVC-Pb<sub>3</sub>O<sub>4</sub> nanocomposite films. FTIR analysis shows the small shift in 650, 845 and 1732 cm<sup>-1</sup> band positions as a confirmation of interaction between Pb<sub>3</sub>O<sub>4</sub> nanoparticles with PVC polymer matrix. The real permittivity ( $\epsilon_1$ ) decreases with increasing frequency for all samples with the appearance of a relaxation peak at high temperatures. The dielectric loss data ( $\epsilon_2$ ) of the PVC-Pb<sub>3</sub>O<sub>4</sub> nanocomposite revealed a shift of the dielectric absorption peak towards high frequency with increasing the temperature. The activation energy values for both  $\alpha$  and  $\beta$  relaxations almost decreased with increasing the Pb<sub>3</sub>O<sub>4</sub> concentration. The energy density of samples containing Pb<sub>3</sub>O<sub>4</sub> has a lower energy density than the pure PVC polymer film. The exponent  $s$  often increased with increasing the temperature, and this behavior is consistent with overlapping large-polaron tunneling model. The DC activation energy decreased when the percentage of Pb<sub>3</sub>O<sub>4</sub> increased to 3.0 wt% and then increased at 4.0 wt%. Additionally, a convergence between these values and the activation energies of  $\alpha$  and  $\beta$  relaxations observed, which indicates that the same type of charge carriers participate in the processes.

**Keywords:** PVC nanocomposites; Dielectric constant; Energy density; Activation energy

## 1. Introduction

Many researchers have recently been interested in studying polymer nanocomposites because of their excellent properties <sup>[1,2]</sup>. Studies of polymer nanocomposites grafted with low percentages of fillers have shown distinct properties over conventional composites <sup>[3-5]</sup>. It appears from the relationship between permittivity and frequency of the polymer that it does not respond immediately upon application of an electric field. The measurement of thermally stimulated depolarization currents used to characterize the segmental mobility and interfacial structures in materials <sup>[6]</sup>. It is known that inorganic materials possess large permittivity, but suffer from relatively small breakdown strength and mechanical properties due to high sintering temperature and porosity. Although organic polymers possess higher breakdown strength, excellent mechanical properties and handling, they suffer from smaller permittivity <sup>[7]</sup>. New composite materials were produced through the mixing of inorganic nanoparticles with the polymer matrix and thus developed enhanced dielectric and energy storage properties <sup>[8]</sup>.

One of the distinguished polymers is the polyvinyl chloride (PVC) polymer because of its high performance and low cost. Therefore, PVC is involved in many applications such as pipes, medical devices and insulation cables <sup>[9]</sup>.  $\text{Pb}_3\text{O}_4$  ceramic material has a high dielectric constant 13 -17 as reported in the literature <sup>[10]</sup>. Previous studies investigated the dielectric properties of PVC/inorganic nanoparticle composites. For instance, the addition of  $\text{Cr}_2\text{O}_3$  nanoparticles to PVC polymer by Hassen et al. led to an increase in the dielectric permittivity and AC conductivity <sup>[11]</sup>. In El Sayed's work, the dielectric permittivity of PVC was enhanced after adding PbO nanoparticles <sup>[12]</sup>. Abouhaswa and Taha investigated the dielectric properties of PVC matrix

upon addition of copper oxide nanoparticles <sup>[13]</sup>. As expected, the dielectric permittivity of PVC nanocomposites improved by addition of nano-CuO particles. Ahmed et al. studied the dielectric properties of graphene nanoplatelet fillers in PVC composites <sup>[14]</sup>. In their case, dielectric permittivity was also increased by adding the graphene nanoplatelets. Ramazanov and Rahimli investigated dielectric properties of TiO<sub>2</sub> based PVC systems by combining various concentrations of TiO<sub>2</sub> nanoparticles <sup>[15]</sup>. As depicted, the dielectric permittivity of composites increased at smaller weight fractions but decreased at higher loadings. In contrast, the dielectric constant of PVC polymer films decreased after adding La<sub>0.95</sub>Bi<sub>0.05</sub>FeO<sub>3</sub> nanoparticles <sup>[16]</sup>.

This work is an extension of our previous research in which we have studied optical and TGA analyses for PVC-Pb<sub>3</sub>O<sub>4</sub> polymer nanocomposites <sup>[17]</sup>. Therefore, we are currently studying the structure of the prepared nanocomposites via FTIR measurements within frequency range (400 – 4000 cm<sup>-1</sup>). Finally, the broadband dielectric properties of these nanocomposite films were studied at a frequency from 0.1 Hz to 20 MHz at temperatures (303 – 383 K).

## **2. Experimental details**

The polymer nanocomposite films of PVC-Pb<sub>3</sub>O<sub>4</sub> with different Pb<sub>3</sub>O<sub>4</sub> content prepared by the solution mixing process. In the procedure, 2.0 g PVC powder dissolved in 40 ml THF with stirring for 60 min at 300 K. Then a clear solution obtained and 0.01, 0.03, and 0.04 g of Pb<sub>3</sub>O<sub>4</sub> nanoparticles added with stirring for 1h <sup>[17]</sup>. The polymer nanocomposite solution was poured into a glass dish and dried in the air for 24 h. Finally, polymer films peeled off the glass dish and cut into squares.

JASCO FT/IR-6100 spectrometer was used to measure the FTIR spectra of the PVC-Pb<sub>3</sub>O<sub>4</sub> nanocomposite films within the frequency range 400–4000 cm<sup>-1</sup>. The frequency-dependent dielectric parameters measured using Novocontrol spectrometer within a frequency range from 0.1 Hz - 20 MHz at ambient temperatures from 303 to 383 K with a voltage amplitude of 1 V.

### 3. Results and discussion

The measured FTIR spectra of PVC-Pb<sub>3</sub>O<sub>4</sub> nanocomposite films are displayed in Fig.1.

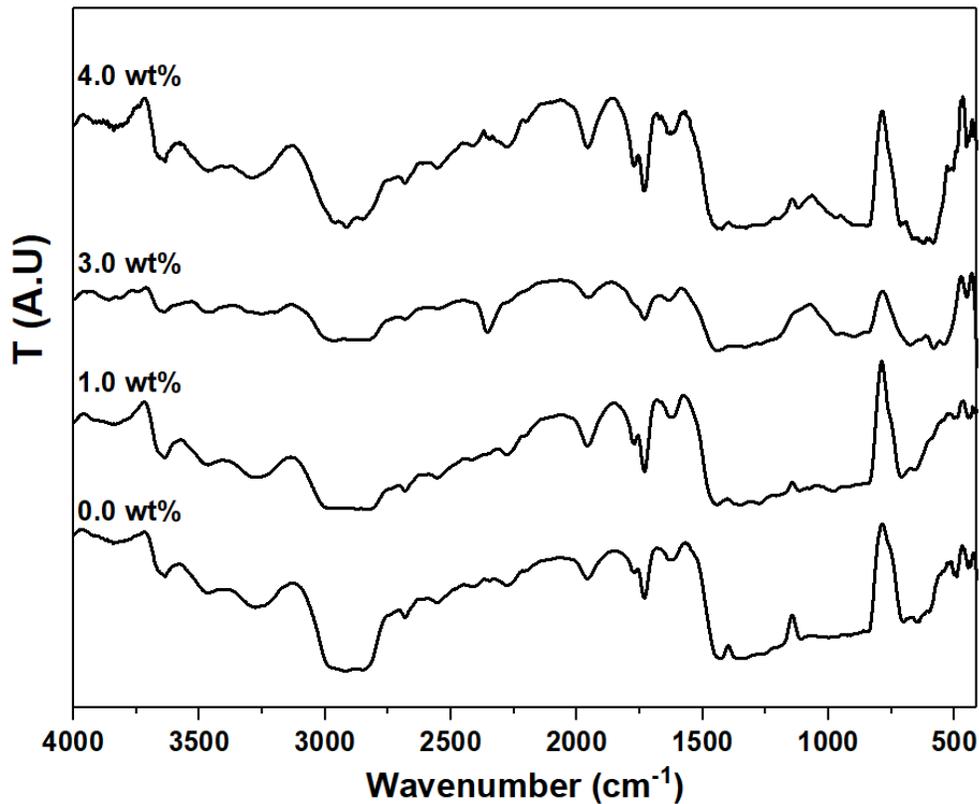


Fig.1. Measured FTIR spectra of PVC films doped with 0, 1, 3 and 4 wt% Pb<sub>3</sub>O<sub>4</sub>

The absorption bands in the infrared spectrum of polyvinyl chloride at 501, 650 and 700 cm<sup>-1</sup> are assigned to the amorphous absorption band of C–Cl

stretching, C–Cl crystalline absorption band and isotactic C–Cl stretching, respectively <sup>[18]</sup>. The band around 845 attributed to  $\nu(\text{C–C})$  stretching vibrations. The bands at 1110 and 1187  $\text{cm}^{-1}$  correspond to perpendicular chain stretch and parallel chain stretch. The absorption band at 1357  $\text{cm}^{-1}$  corresponds to  $\text{CH}_2$  wag and the other band at 1425  $\text{cm}^{-1}$  is due to the bending mode of  $\text{CH}_2$ . The absorption band at 1624  $\text{cm}^{-1}$  is assigned to the carbon-carbon double bond stretching vibration for conjugated bonds, or either aromatic or aliphatic or both. The band at 1732  $\text{cm}^{-1}$  is probably from the carbonyl stretching vibration. The wideband at 2915  $\text{cm}^{-1}$  corresponds to the  $\text{CH}_2$  asymmetric stretching mode, the peak broadening is due to the intermolecular and intra-molecular hydrogen bonds. While the band of OH stretching appeared at 3462  $\text{cm}^{-1}$  <sup>[19]</sup>. For PVC film containing 1.0, 3.0 and 4.0 wt.% of  $\text{Pb}_3\text{O}_4$  nanoparticles, the same absorption bands are observed. However, the small shift in 650, 845 and 1732  $\text{cm}^{-1}$  band positions confirmed the interaction between  $\text{Pb}_3\text{O}_4$  nanoparticles with PVC polymer matrix.

The complex dielectric constant describe the phenomena of dielectric dispersion through the following relation <sup>[20]</sup>

$$\epsilon^* = \epsilon_1 - i\epsilon_2 \quad (1)$$

where  $\epsilon_1$  is the real permittivity and imaginary part  $\epsilon_2$  is known as dielectric loss. Frequency dependence real dielectric permittivity ( $\epsilon_1$ ) for PVC- $\text{Pb}_3\text{O}_4$  nanocomposite films at various temperatures presented in **Fig.2**. It is obvious that the higher the temperature, the better the dielectric constant of all the films.

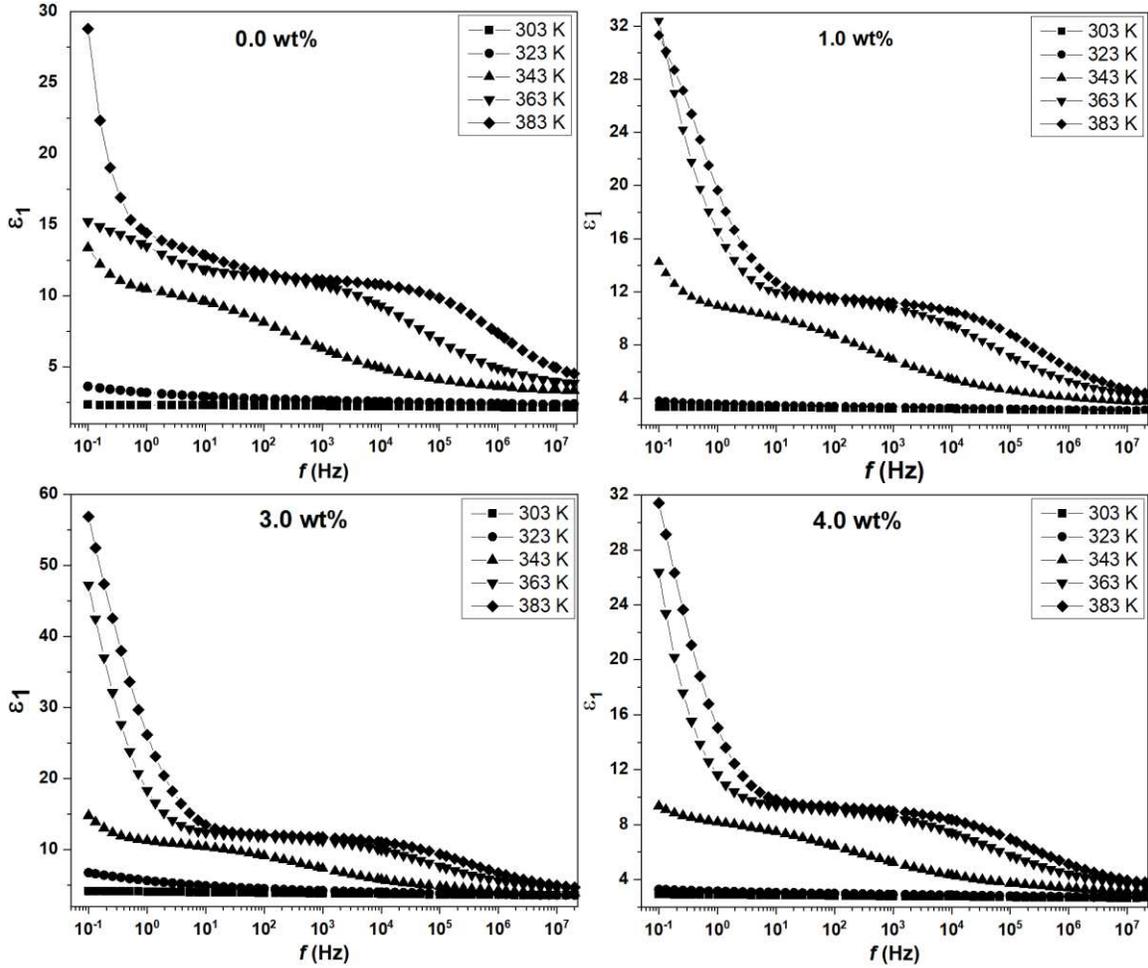


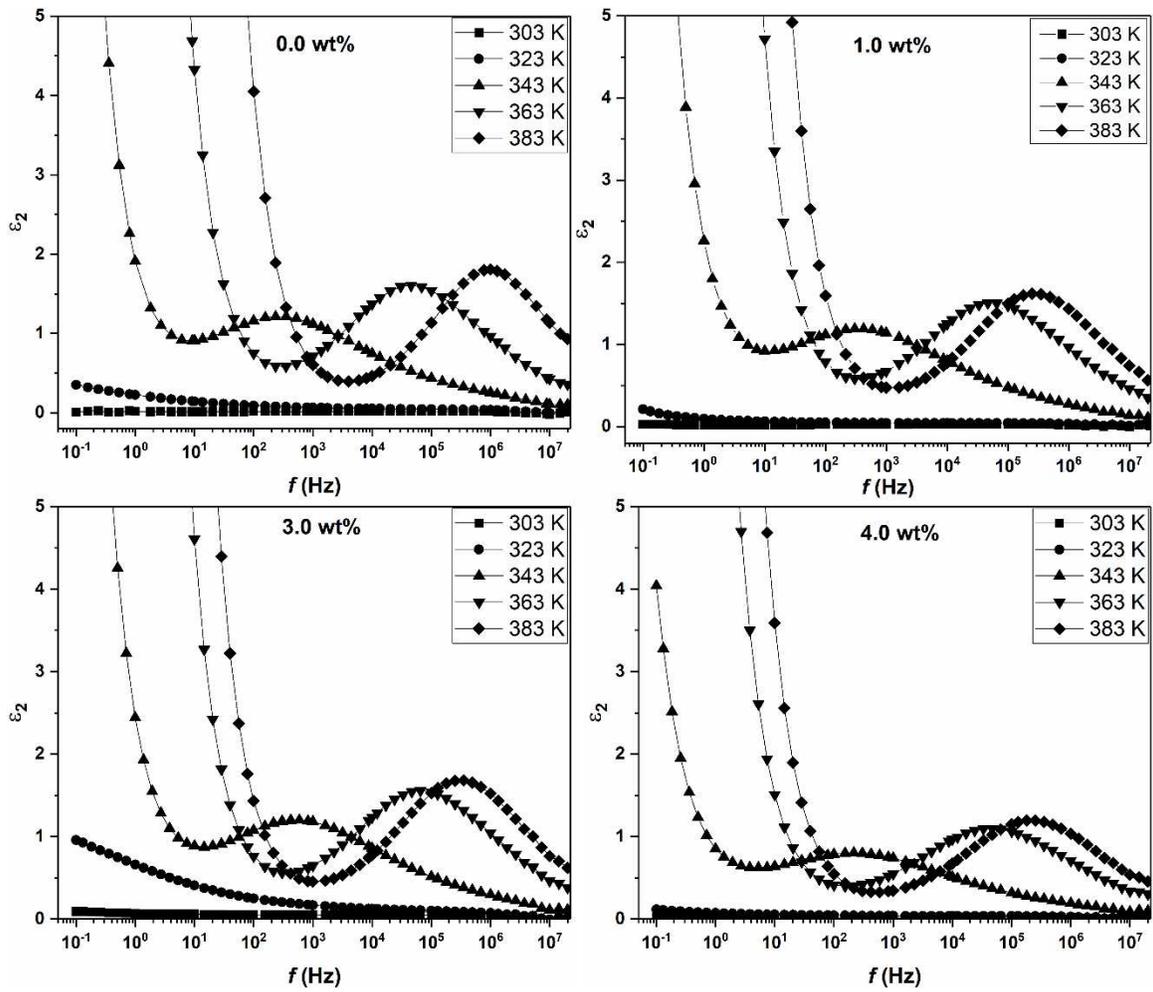
Fig.2. Measured real dielectric permittivity at different temperatures for the PVC-Pb<sub>3</sub>O<sub>4</sub> films

Fig.2 shows the effect of increasing the permittivity due to an increase in temperature from 303 to 383 K. We also noticed from the figure that the real permittivity decreases with increasing frequency for all samples with the appearance of a relaxation peak at high temperatures. The decrease in real permittivity with increasing the frequency is explained by the decrease of space charge polarization. As the space charge polarization increased at a lower frequency, a potential barrier was generated and the charges accumulated at the grain boundary to increase the real permittivity [20].

The dielectric loss data ( $\epsilon_2$  vs.  $f$ ) of the PVC-Pb<sub>3</sub>O<sub>4</sub> nanocomposites (**Fig.3**) indicates that the dielectric loss values are observed to be increasing more rapidly at low frequency. On further inspection of **Fig.3**, one may notice a shift of the dielectric absorption peak towards high frequency with increasing the temperature. The Arrhenius activation model is the simplest one that is applied to describe the temperature dependence of relaxation times through the condition  $\omega_{\max}\tau = 1$ . Thus the Arrhenius temperature behavior of relaxation frequency given by [21]:

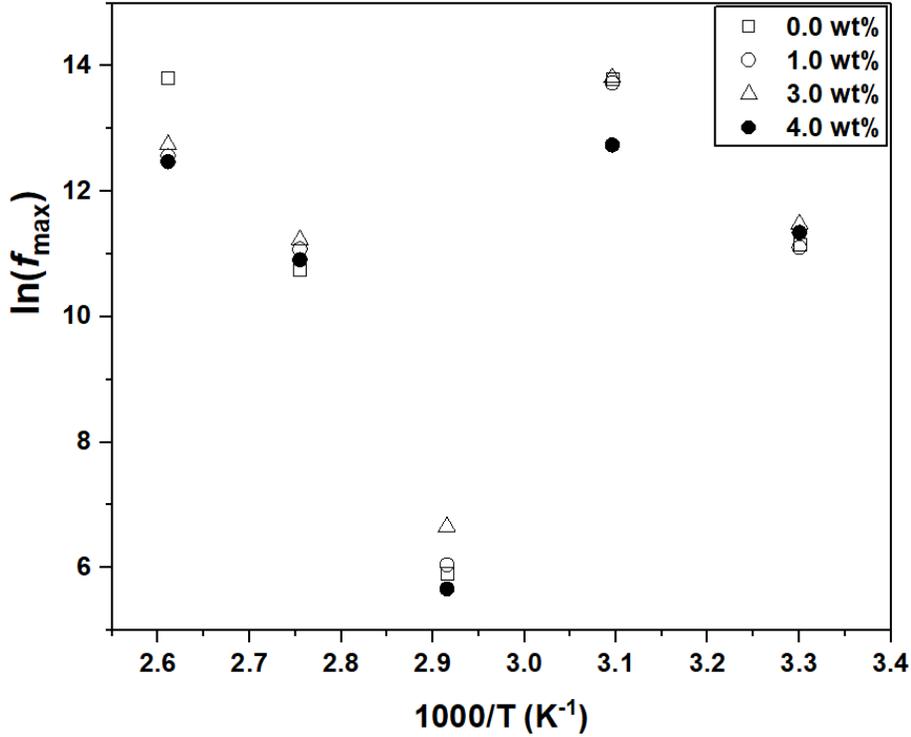
$$f_{\max} = f_o e^{\left(\frac{-E_a}{k_\beta T}\right)} \quad (2)$$

where  $f_o$  is constant,  $E_a$  the activation energy and  $k_\beta$  Boltzmann's constant. From the plots of  $\ln(f_{\max})$  vs.  $1000/T$ , which are linear, values for the apparent activation energies are derived (**Fig.4**).



**Fig.3.** Plot of  $\epsilon_2$  vs. frequency for PVC-Pb<sub>3</sub>O<sub>4</sub> nanocomposite films

In **Table.1** the values of the activation energy for  $\beta$  and  $\alpha$  relaxations were recorded for the different nanocomposite films. In general, the activation energy values for both  $\alpha$  and  $\beta$  relaxations decreased with increasing the Pb<sub>3</sub>O<sub>4</sub> concentration.



**Fig.4.** Plots of  $\ln(f_{\max})$  versus  $1000/T$  for the PVC- $\text{Pb}_3\text{O}_4$  nanocomposite films

**Table.1.** Activation energies of the PVC-- $\text{Pb}_3\text{O}_4$  nanocomposite films

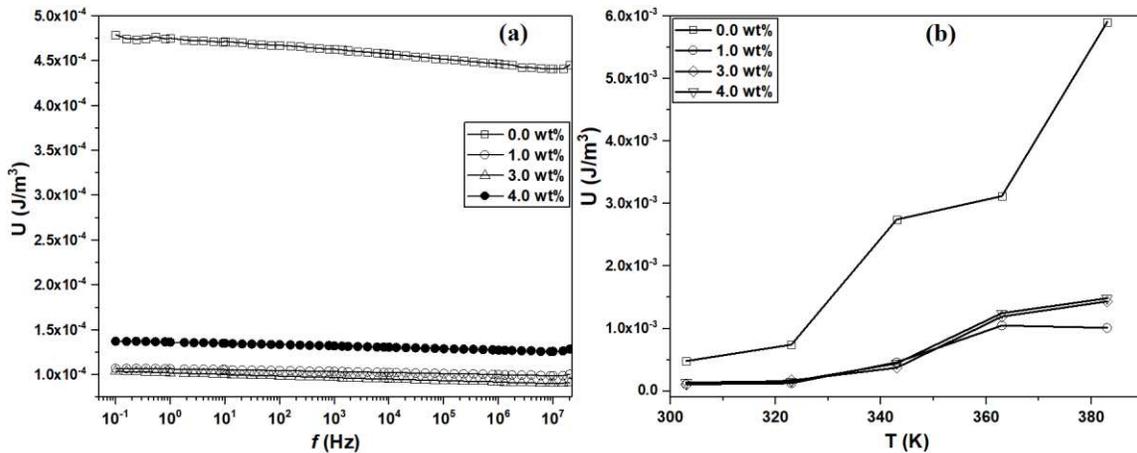
$\text{Pb}_3\text{O}_4$ content	$E_a$ (eV)	
	$\beta$	$\alpha$
0.0 wt%	1.11	2.25
1.0 wt%	1.10	1.86
3.0 wt%	0.98	1.74
4.0 wt%	0.59	1.94

In polymer composites, the main relaxation  $\alpha$ -transition is related to the glass transition of the polymer and refers to cooperative transitions and the relaxation  $\beta$ -transition closest to the glass transition temperature associate this transition with the motions of short (smaller than a segment) sections of the

main chain, the mobility of the side groups and their fragments. In connection with this, as the ratio of  $\text{Pb}_3\text{O}_4$  increased in the PVC polymer matrix, the activation energies for rotational and translational movements of segments and cooperative modes of segmental mobility decrease. One can estimate the corresponding gain in energy density (electrostatic energy) for a linear dielectric through the following relationship [22];

$$U = \frac{1}{2} \epsilon_0 \epsilon_1 E^2 \quad (3)$$

where  $U$  is the maximum energy density the dielectric can contain,  $\epsilon_0$  is the permittivity of free space,  $\epsilon_1$  is the relative permittivity of the dielectric and  $E$  is the electric field is proportional to the voltage applied ( $E = V/d$ ). In **Fig.5a**, the energy density is estimated at 303 K as a function of  $\text{Pb}_3\text{O}_4$  content. Samples containing  $\text{Pb}_3\text{O}_4$  have a lower energy density than the pure PVC polymer film. This result could be due to the decrease of real permittivity ( $\epsilon_1$ ) and increased dielectric loss ( $\epsilon_2$ ). As the temperature increase, the energy density increase for all nanocomposite films (**Fig.5b**). This increase in the energy density is due to the high mobility of large polymer chains and interfacial polarization [23].

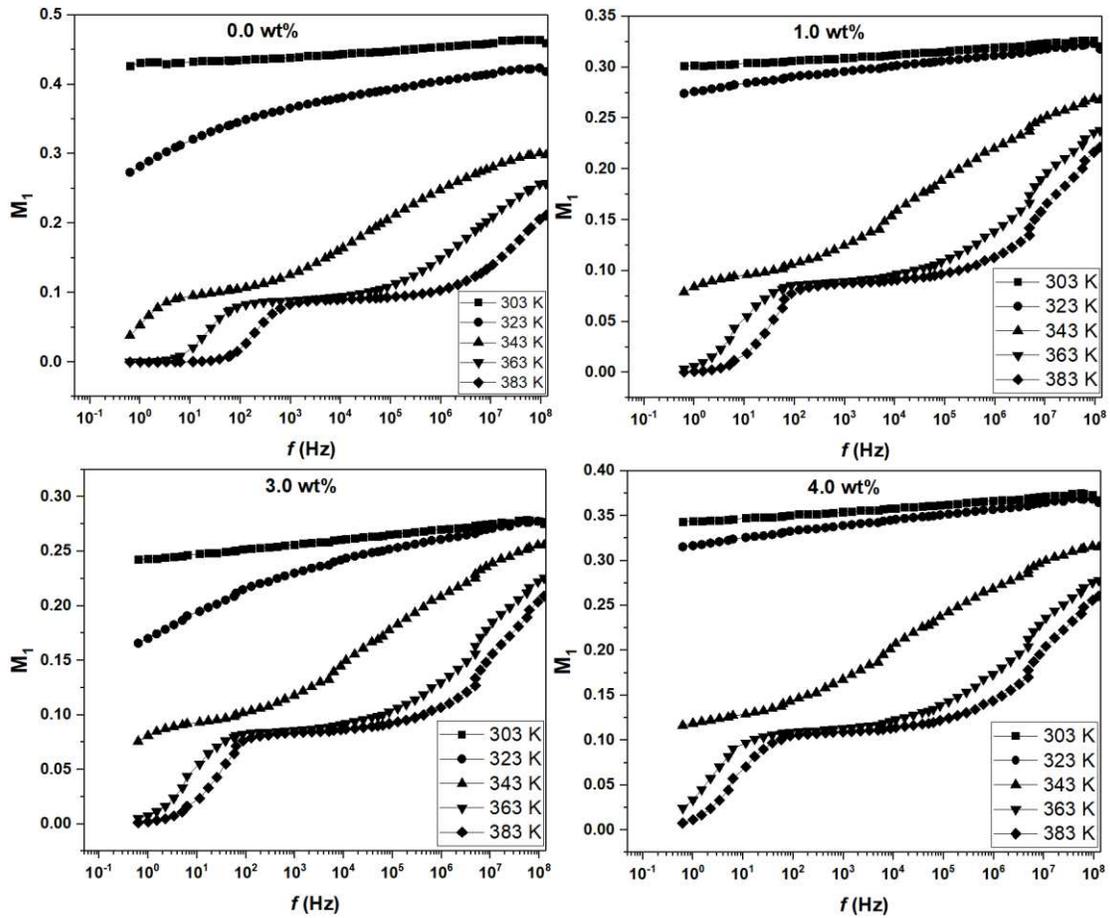


**Fig.5.** Variation of energy density (U) with (a) frequency at 303 K and (b) temperature at 0.1 Hz for all nanocomposite films

The electric modulus analysis can describe relaxation phenomena in different materials. The expression for the electric modulus ( $M^*$ ) is given by [24];

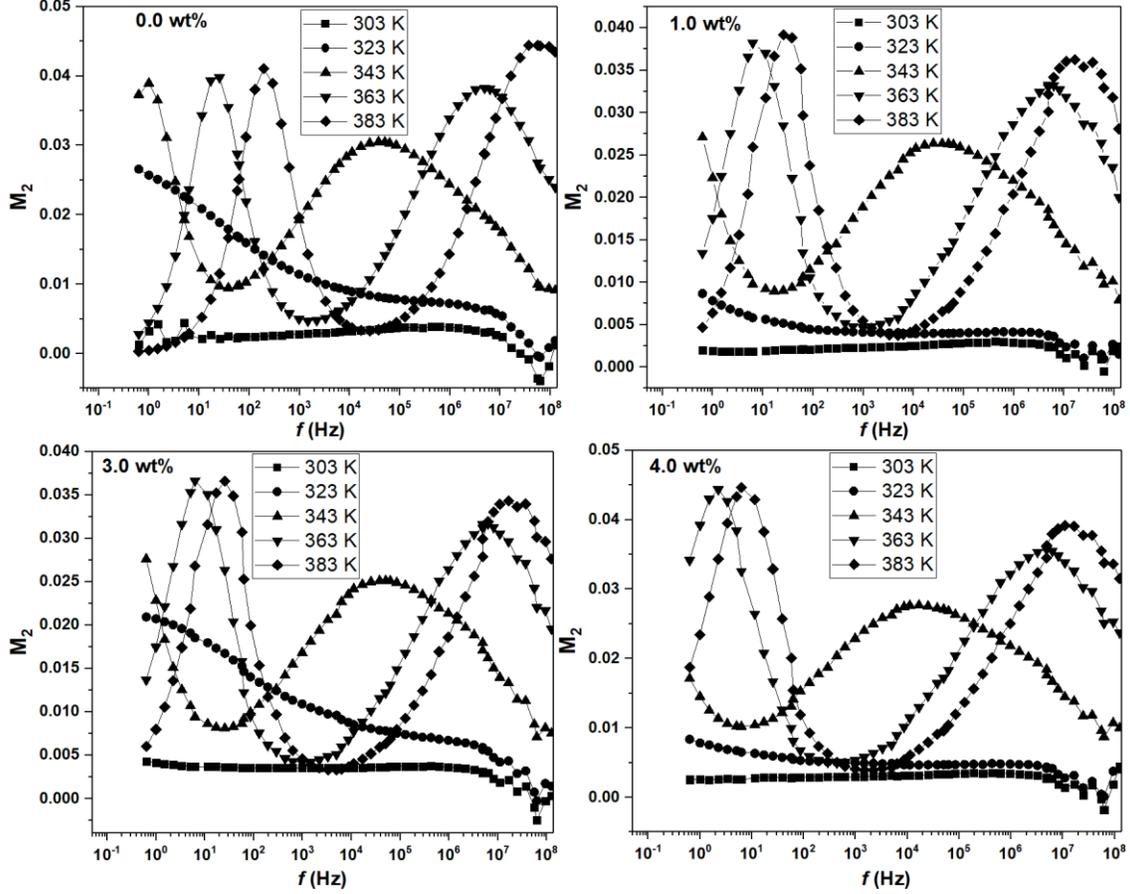
$$M^* = \frac{1}{\varepsilon^*} = \frac{1}{\varepsilon_1 - j\varepsilon_2} = \frac{\varepsilon_1}{\varepsilon_1^2 + \varepsilon_2^2} + j \frac{\varepsilon_2}{\varepsilon_1^2 + \varepsilon_2^2} = M_1 + jM_2 \quad (4)$$

Where  $M_1$  defines the real electric modulus and  $M_2$  is the imaginary part electric modulus. We can see in **Fig.6** that at low frequencies, the values of  $M_1$  are lowered as the temperature rises in all nanocomposite films. This comes due to reducing the contribution of electrode polarization [25]. We also notice an increase in  $M_1$  with increasing frequency at all temperatures.



**Fig.6.** Plots of  $M_1$  versus frequency at different temperatures for the PVC- $Pb_3O_4$  nanocomposite films

The imaginary part of electric modulus ( $M_2$ ) for the PVC- $Pb_3O_4$  nanocomposite films presented in **Fig.7**. At low frequency, the electrode polarization is suppressed with increasing the temperature. At high temperatures, two relaxation processes are depicted and shifted to the high-frequency side.



**Fig.7.** Graphs of  $M_2$  versus frequency at different temperatures for the PVC-Pb<sub>3</sub>O<sub>4</sub> nanocomposite films

Additionally, the imaginary electric modulus ( $M_2$ ) related to the frequency ( $\omega$ ) by the modified Kohlrausch-Williams-Watts (KWW) relation [26]:

$$M_2 = \frac{M_2^{max}}{(1+\beta) + \frac{\beta}{1+\beta} \left[ \beta \left( \frac{\omega_{max}}{\omega} \right) + \left( \frac{\omega}{\omega_{max}} \right)^\beta \right]} \quad (5)$$

Where  $M_2^{max}$  denotes the maximum value of  $M_2$  at  $\omega_{max}$  and  $\beta$  is the KWW parameter. After fitting the graphs in **Fig.7** with **eq.5**, the values of  $\beta$  are evaluated and recorded in **Table.2**.

**Table.2.** The evaluated  $\beta$  values for the PVC--Pb<sub>3</sub>O<sub>4</sub> nanocomposite films

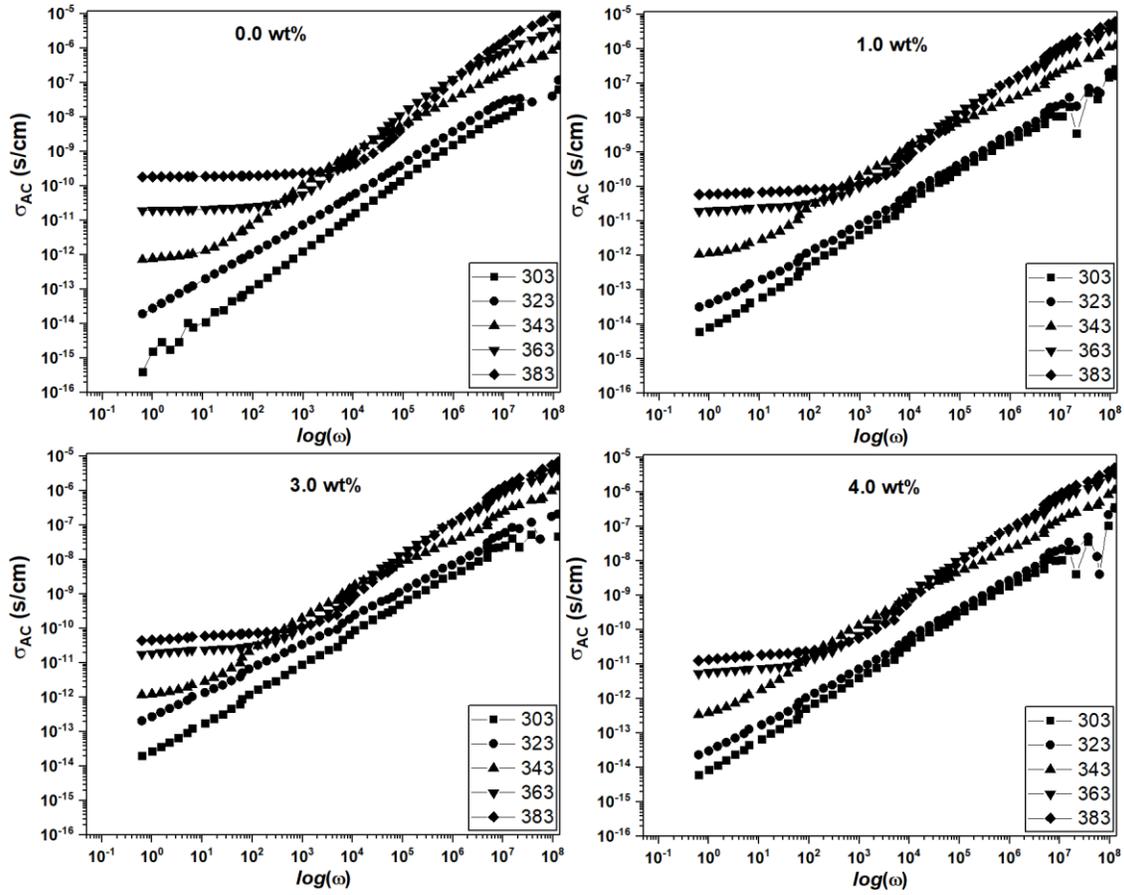
<b>Pb<sub>3</sub>O<sub>4</sub> percentage</b>	<b>303 K</b>	<b>323 K</b>	<b>343 K</b>	<b>463 K</b>	<b>483 K</b>
0.0 wt%	0.62	0.55	0.31	0.45	0.32
1.0 wt%	0.46	0.50	0.26	0.43	0.57
3.0 wt%	0.59	0.50	0.26	0.41	0.57
4.0 wt%	0.59	0.50	0.24	0.40	0.50

As shown in **Table.2**, the estimated values of  $\beta$  less than 1.0, which correlated with non-Debye relaxation [27],

The frequency-dependent conductivity spectra described previously by the following Jonscher power law [28],

$$\sigma_{AC} = \sigma_{DC} + A\omega^s \quad (6)$$

Where A and s are temperature-dependent parameters. This procedure allows analyzing directly the temperature-activated behavior of conduction. Such analysis is useful to reveal the nature of charge species participating in conduction, where the exponent s lies between  $0 < s < 1$ .



**Fig.8.** Plots of  $\sigma_{AC}$  versus frequency at different temperatures for the PVC- $Pb_3O_4$  nanocomposite films

**Table.3.** The estimated values of  $s$  for PVC-- $Pb_3O_4$  nanocomposite films

	<b>Pb<sub>3</sub>O<sub>4</sub> percentage</b>	<b>303 K</b>	<b>323 K</b>	<b>343 K</b>	<b>463 K</b>	<b>483 K</b>
	0.0 wt%	0.99	0.90	0.76	0.75	0.99
	1.0 wt%	0.82	0.84	0.73	0.83	0.98
	3.0 wt%	0.78	0.81	0.76	0.88	0.99
	4.0 wt%	0.81	0.87	0.82	0.85	0.92

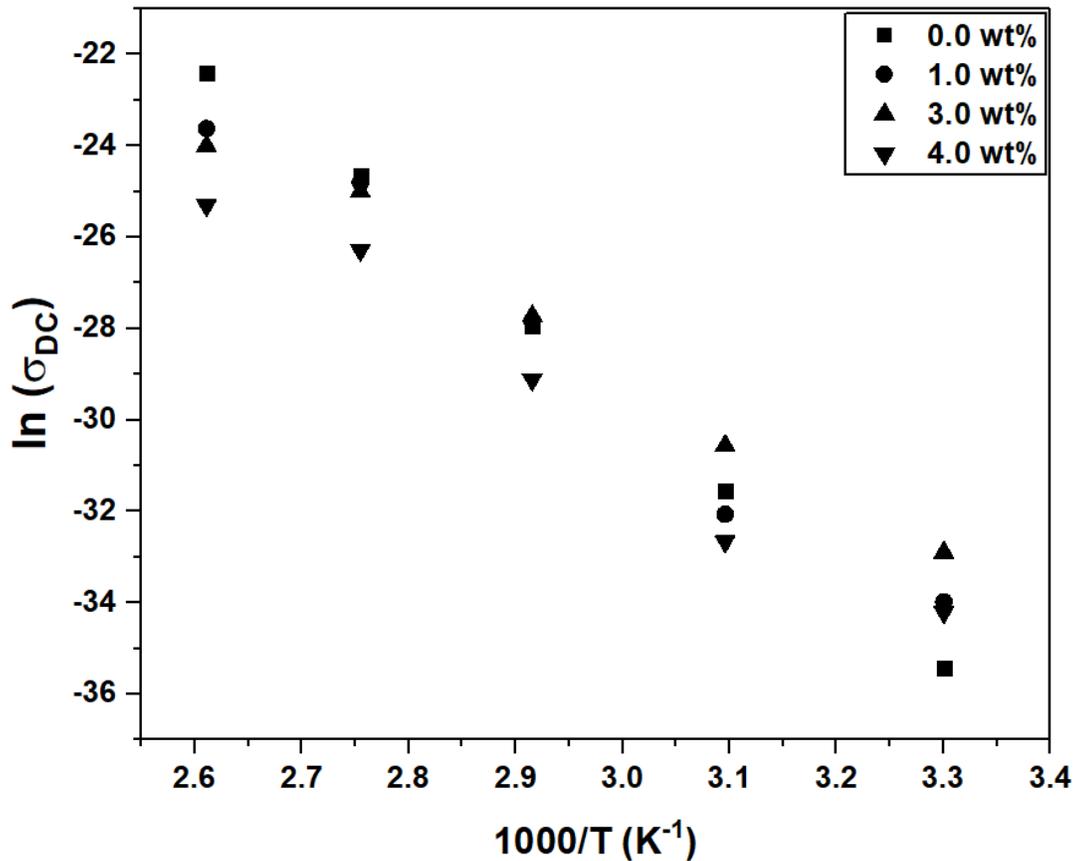
The values of exponent  $s$  are evaluated after fitting the experimental data with **eq.6**. As shown in **Table.3**, the exponents often increased with increasing the

temperature, and this behavior is consistent with overlapping large-polaron tunneling model [29].

The DC conductivity  $\sigma_{DC}$  of a given sample is calculated as the plateau value of ( $f$ ) at low frequency (0.1 Hz) in **Fig.8**. For further analysis, the extrapolated values of  $\sigma_{DC}$  usually plotted as a function of temperature as an Arrhenius plot (**Fig.9**), which is the plot of the  $\ln(\sigma_{DC})$  as a function of the inverse temperature ( $1000/T$ ). The Arrhenius temperature behavior of conductivity is given by [30]:

$$\sigma_{DC} = B e^{\frac{-E_a}{k_{\beta}T}} \quad (7)$$

Here  $E_a$  is the temperature-independent activation energy,  $k_{\beta}$  is the Boltzmann's constant, B is the pre-exponential factor.



**Fig.9.** Graphs of  $\ln(\sigma_{DC})$  as a function of the inverse temperature ( $1000/T$ ) for the PVC-Pb<sub>3</sub>O<sub>4</sub> nanocomposite films

The values of activation energy obtained from the slope of the straight lines in **Fig.8** using the **eq.7**. Hence, the values were 1.65, 1.40, 1.17 and 1.21 eV for the nanocomposite films at 0.0, 1.0, 3.0 and 4.0 wt% Pb<sub>3</sub>O<sub>4</sub>. We notice a convergence between these values and the activation energies of  $\alpha$  and  $\beta$  relaxations, which is an indication that the same type of charge carriers participate in the processes <sup>[27]</sup>. The DC activation energy also decreased when the percentage of Pb<sub>3</sub>O<sub>4</sub> increased to 3.0 wt% and then increased at 4.0 wt%.

#### **4. Conclusion**

This research deals with a study of the structure as well as dielectric properties of the PVC-Pb<sub>3</sub>O<sub>4</sub> nanocomposite films. FTIR analysis showed a small shift in 650, 845 and 1732 cm<sup>-1</sup> band positions as a confirmation of interaction between Pb<sub>3</sub>O<sub>4</sub> nanoparticles with PVC polymer matrix. Dielectric measurements show that the real dielectric constant ( $\epsilon_1$ ) decreased with increasing frequency for all samples with the appearance of a relaxation peak at high temperatures. Plots of the dielectric loss ( $\epsilon_2$ ) for the PVC-Pb<sub>3</sub>O<sub>4</sub> nanocomposites revealed a shift of the dielectric absorption peak towards high frequency with increasing the temperature. The activation energies for both  $\alpha$  and  $\beta$  relaxations almost decreased with increasing the Pb<sub>3</sub>O<sub>4</sub> concentration. The energy density of samples containing Pb<sub>3</sub>O<sub>4</sub> has a lower energy density than the pure PVC polymer film. The exponent  $s$  often increased with increasing the temperature, and this behavior is consistent with overlapping large-polaron tunneling model. The DC activation energy decreased when the

percentage of  $\text{Pb}_3\text{O}_4$  increased to 3.0 wt% and then increased at 4.0 wt%. Additionally, a convergence between these values and the activation energies of  $\alpha$  and  $\beta$  relaxations observed, which indicates that the same type of charge carriers participate in the processes.

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## Figures

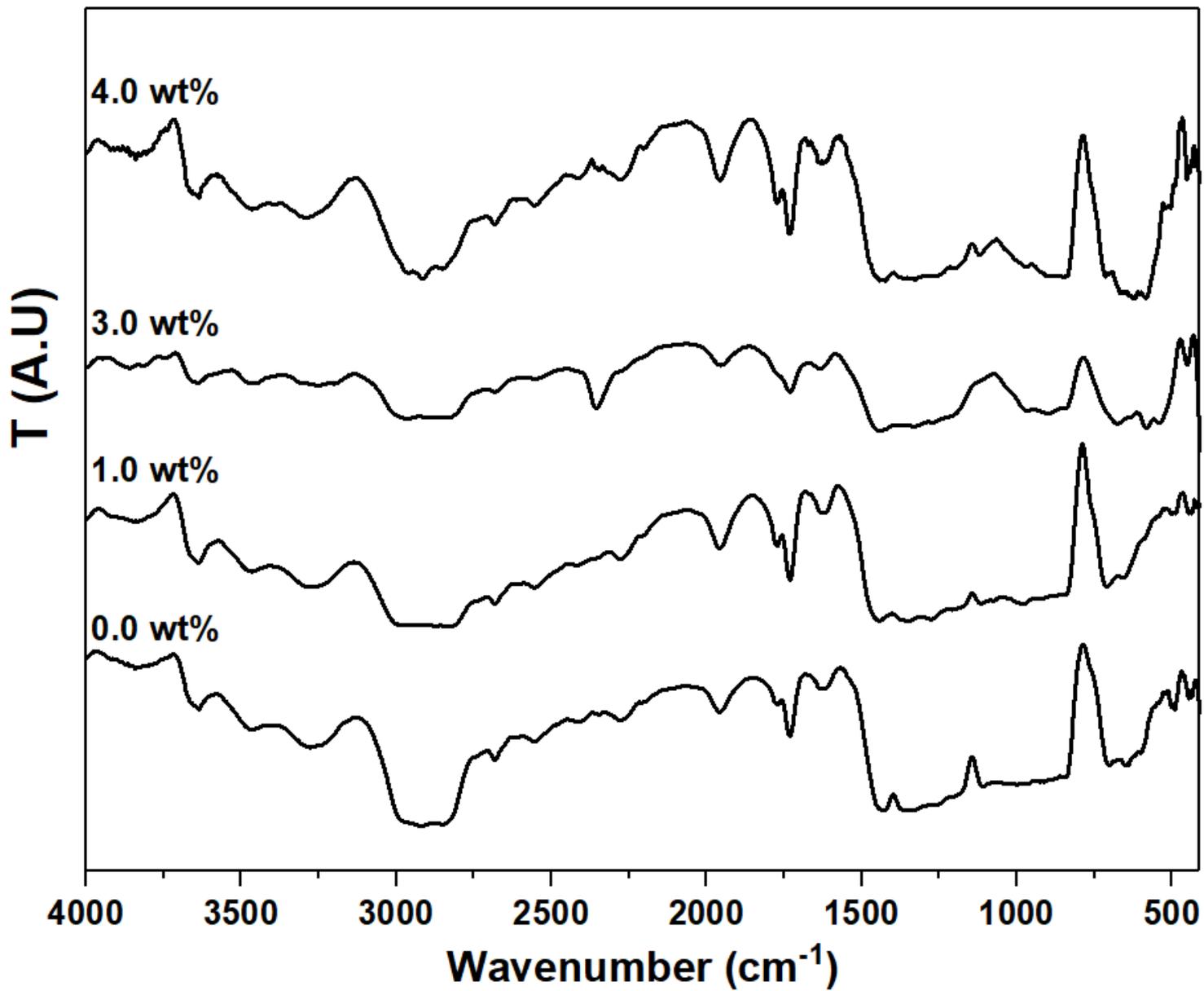


Figure 1

Measured FTIR spectra of PVC films doped with 0, 1, 3 and 4 wt% Pb3O4

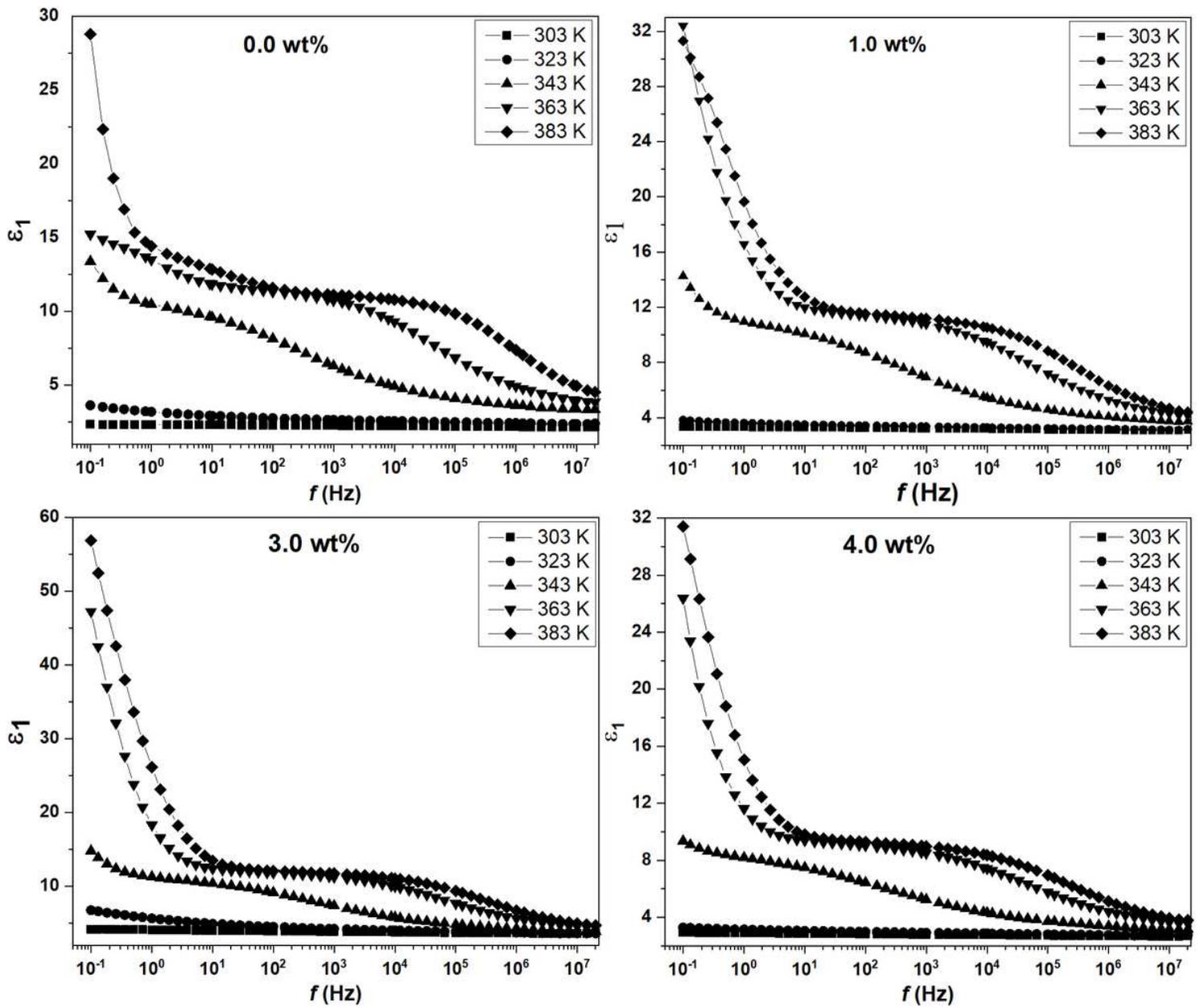


Figure 2

Measured real dielectric permittivity at different temperatures for the PVC-Pb304 films

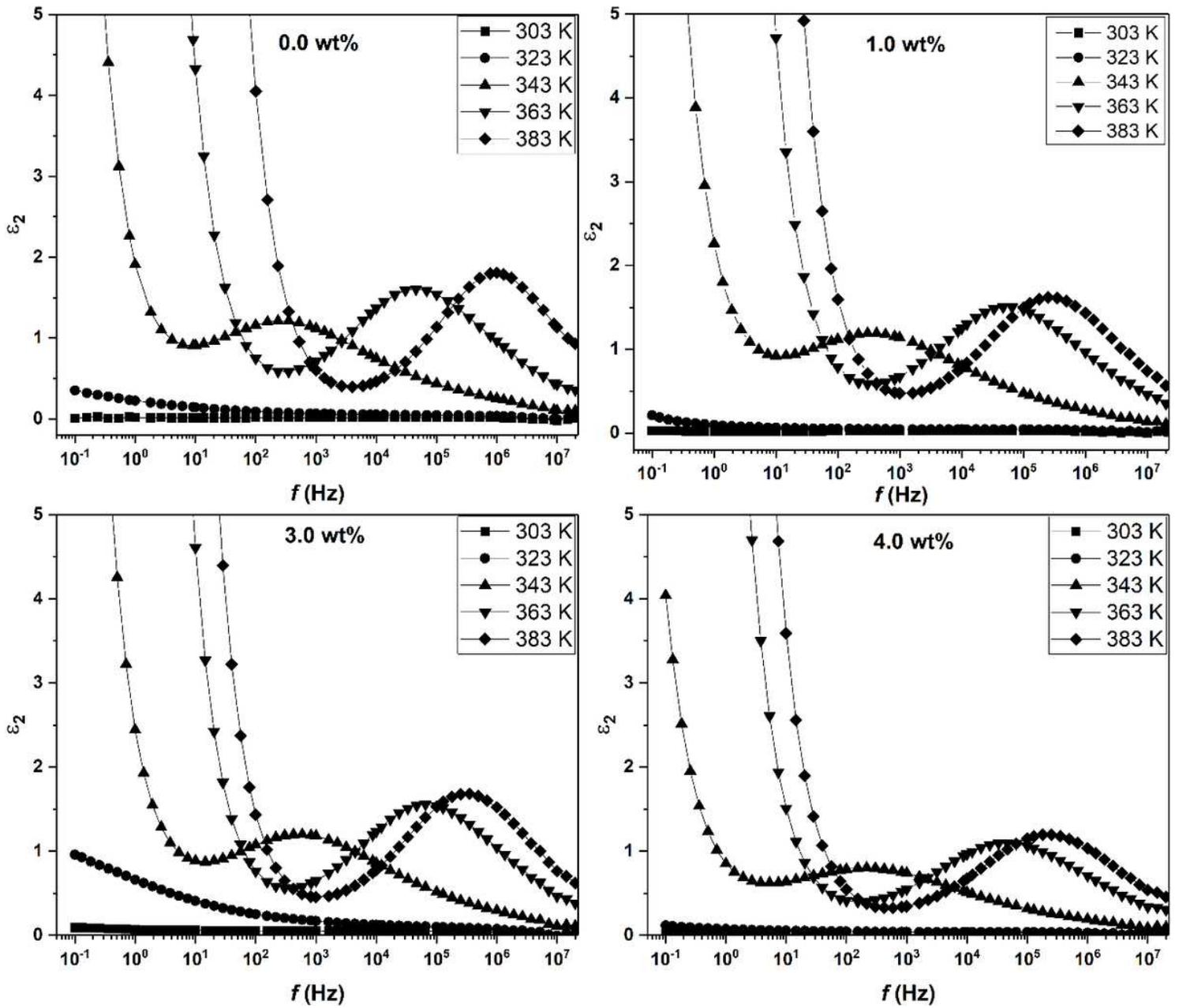


Figure 3

Plot of  $\epsilon_2$  vs. frequency for PVC-Pb304 nanocomposite films

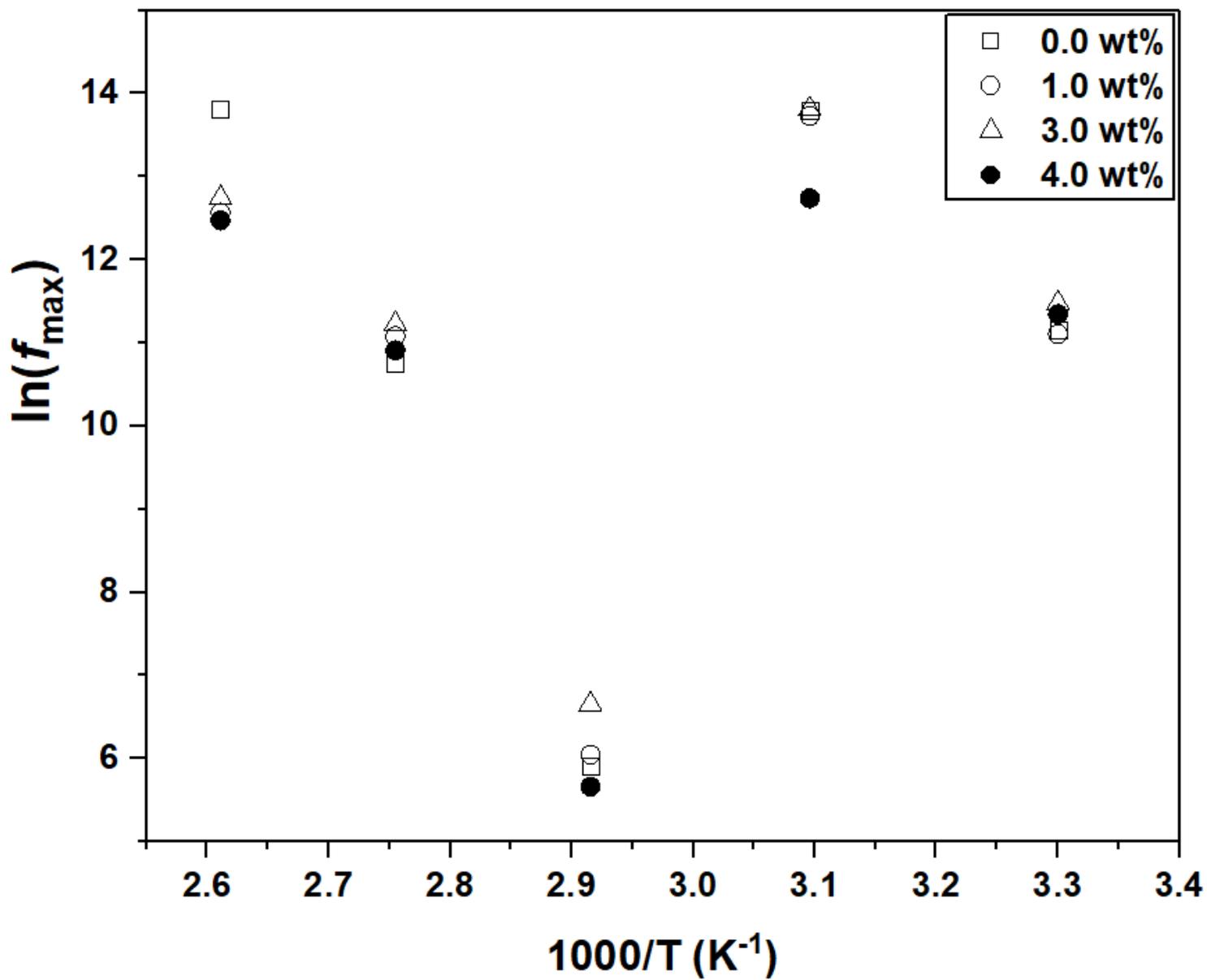
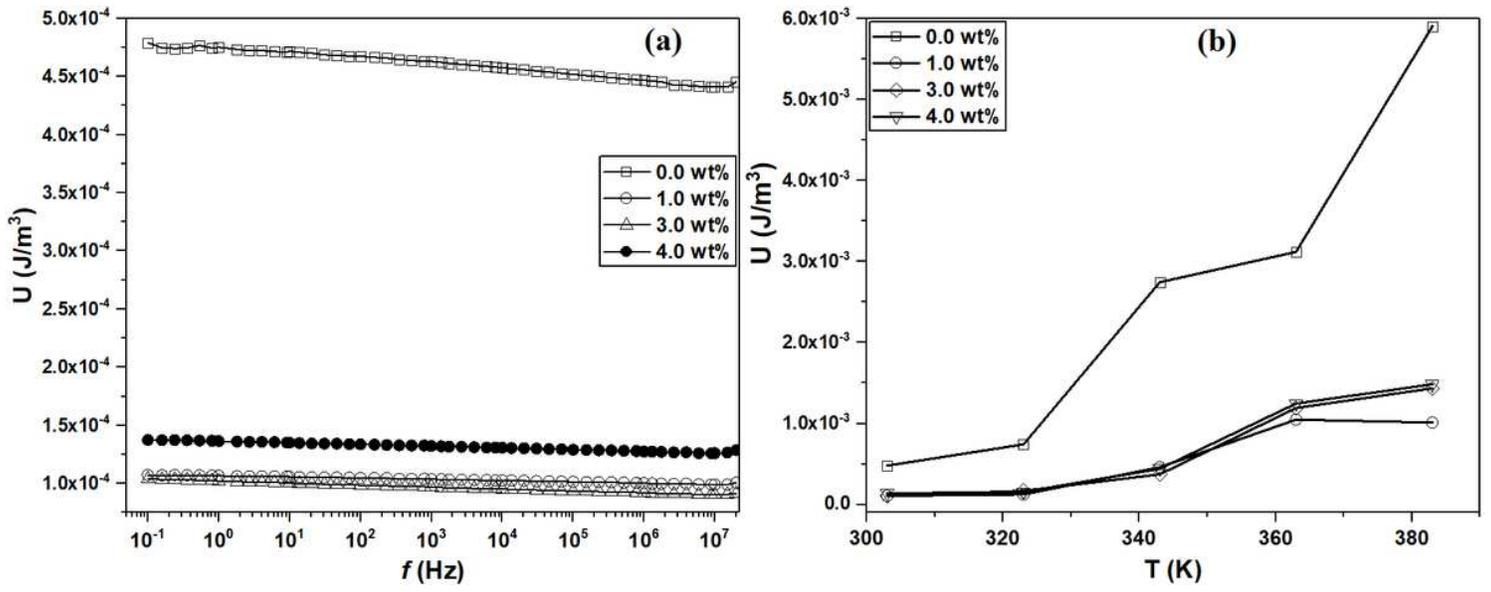


Figure 4

Plots of  $\ln(f_{\max})$  versus  $1000/T$  for the PVC- $\text{Pb}_3\text{O}_4$  nanocomposite films



**Figure 5**

Variation of energy density ( $U$ ) with (a) frequency at 303 K and (b) temperature at 0.1 Hz for all nanocomposite films

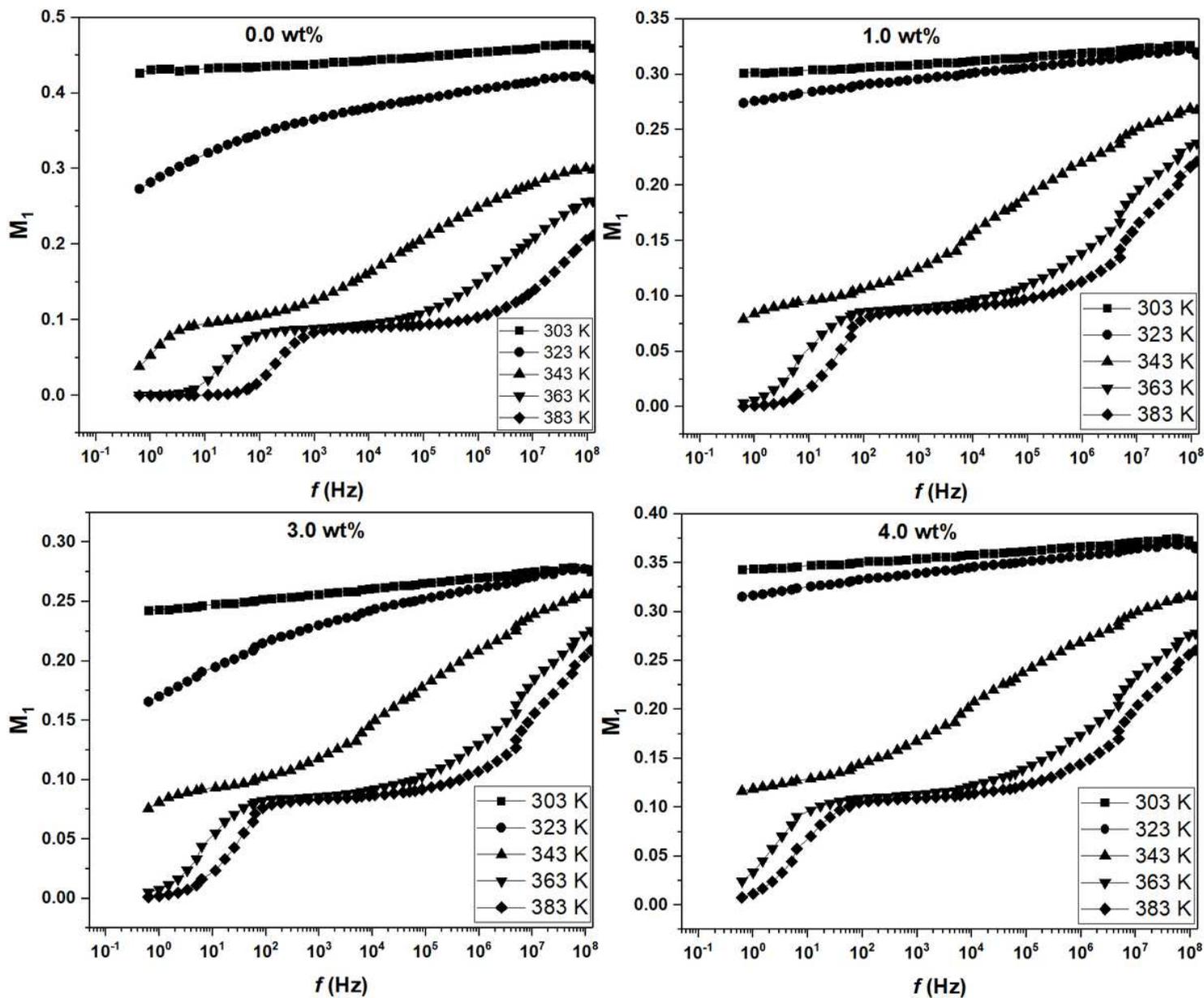


Figure 6

Plots of  $M_1$  versus frequency at different temperatures for the PVC-Pb304 nanocomposite films

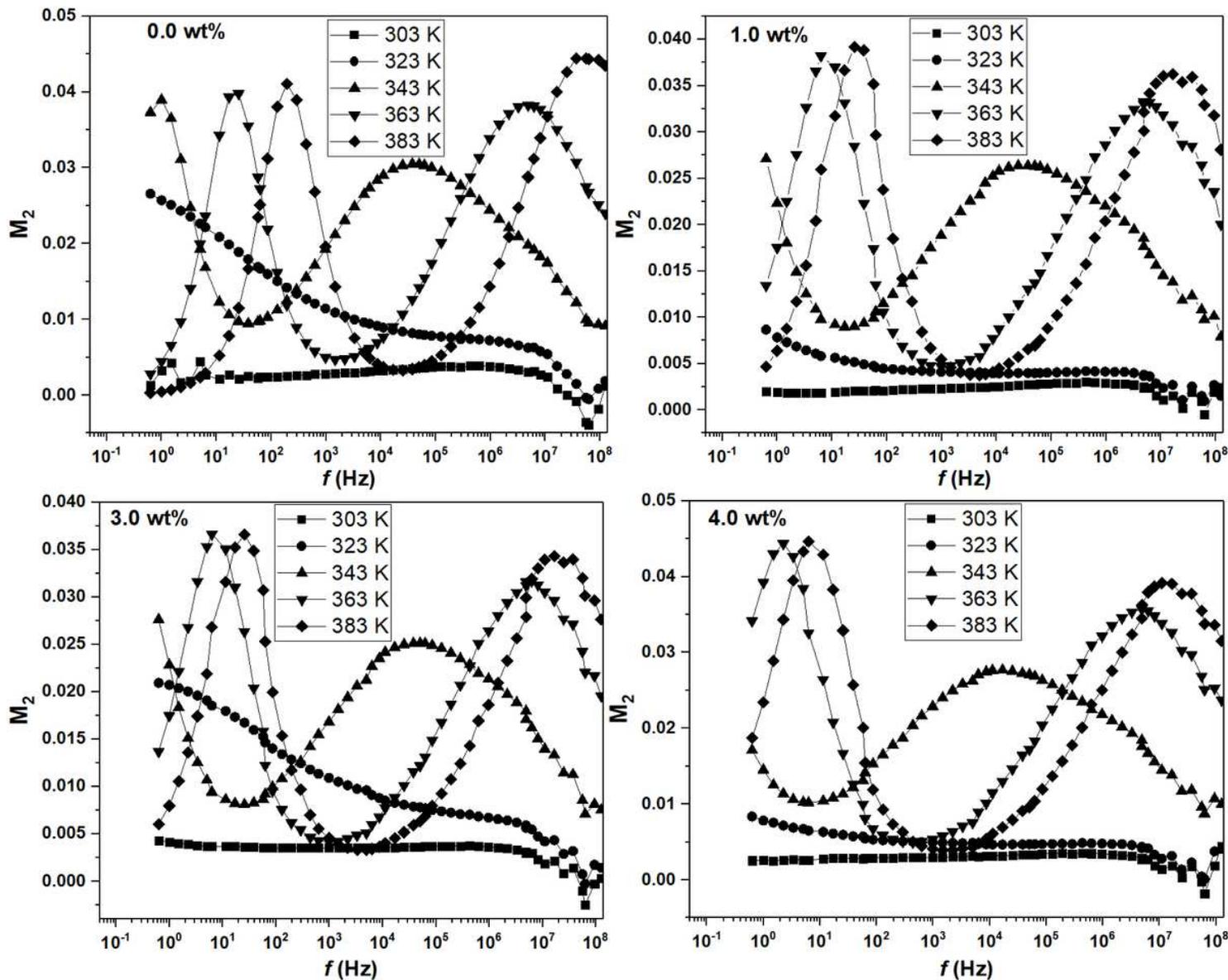


Figure 7

Graphs of  $M_2$  versus frequency at different temperatures for the PVC-Pb304 nanocomposite films

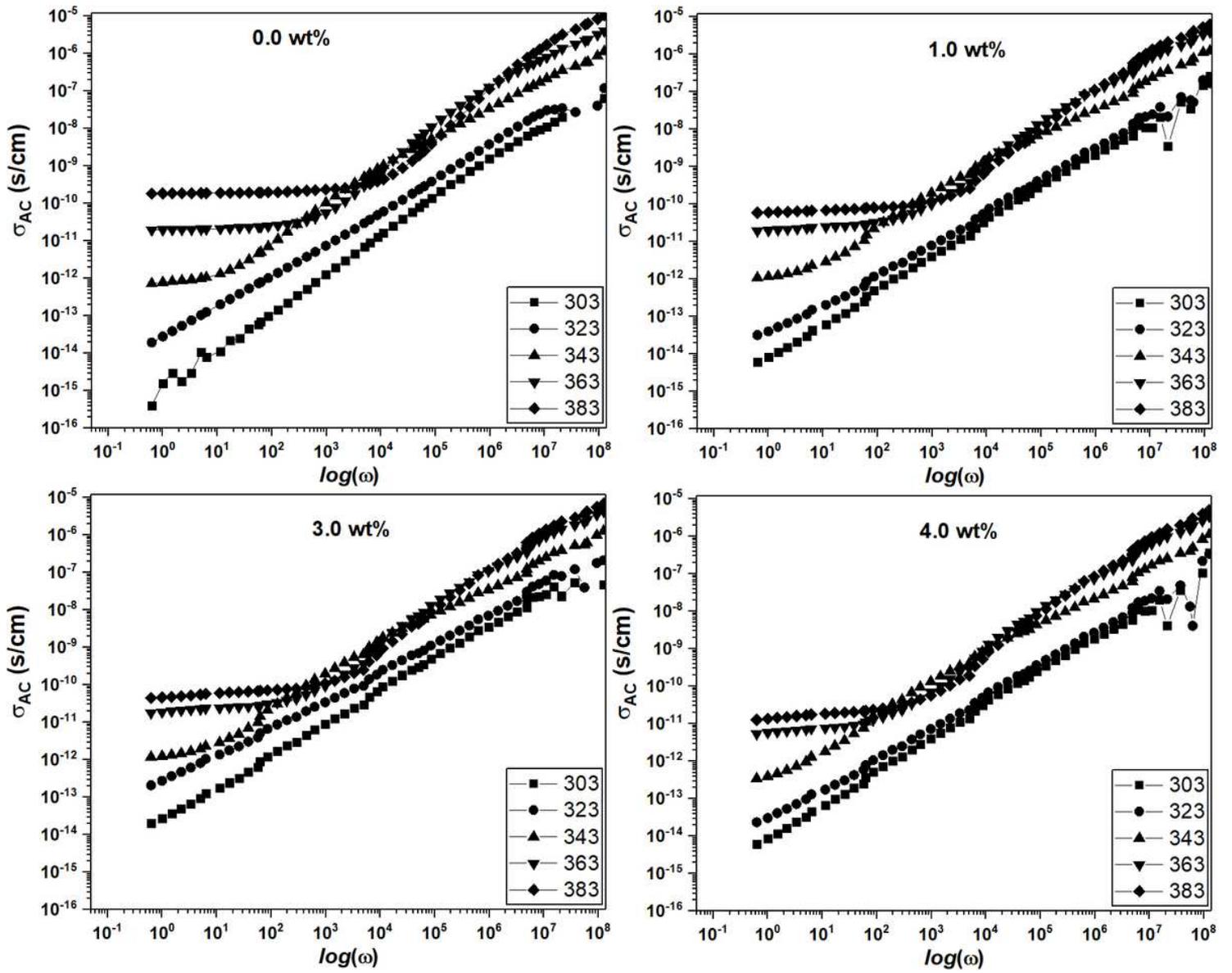


Figure 8

Plots of  $\sigma_{AC}$  versus frequency at different temperatures for the PVC-Pb304 nanocomposite films

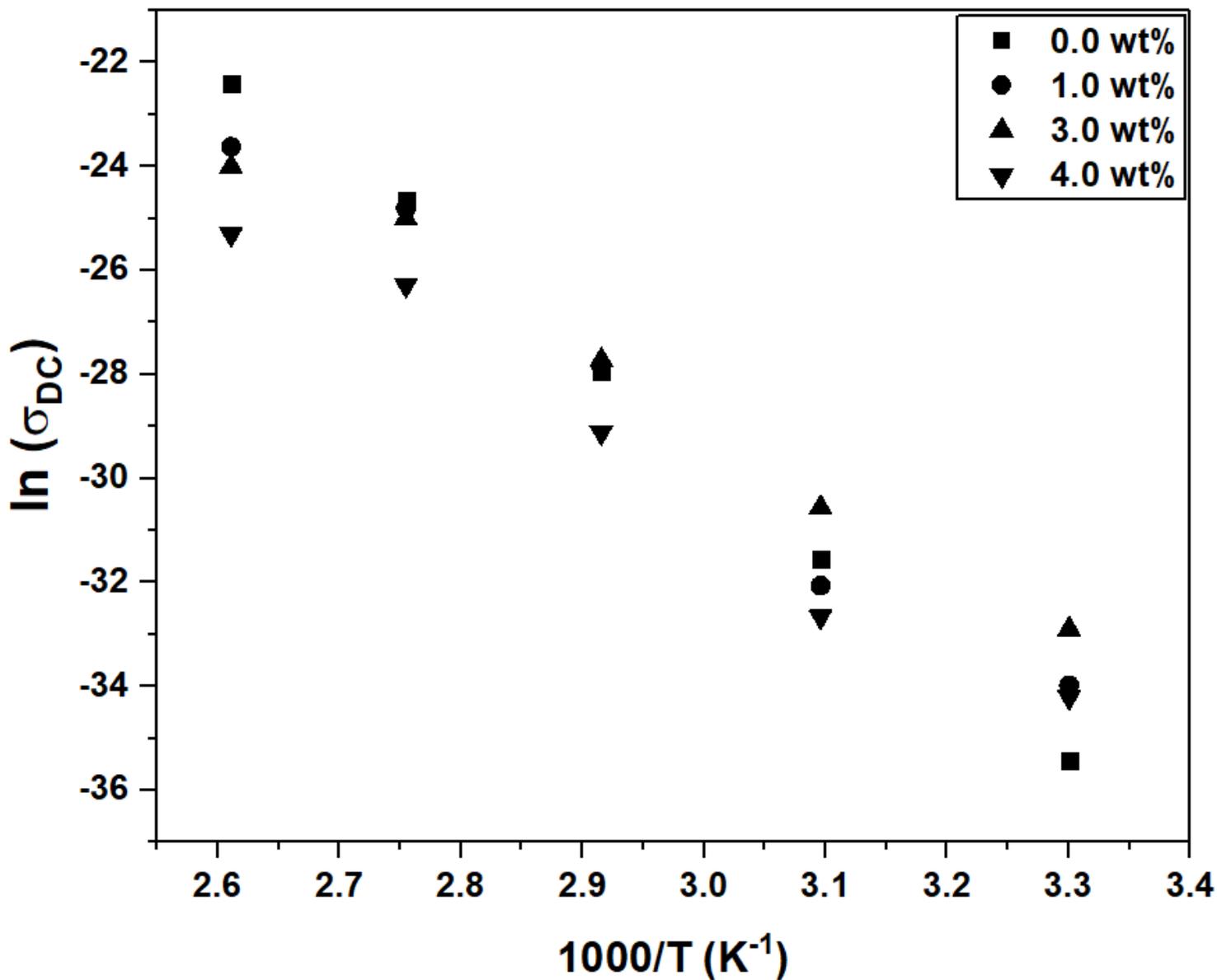


Figure 9

Graphs of  $\ln(\sigma_{DC})$  as a function of the inverse temperature ( $1000/T$ ) for the PVC-Pb304 nanocomposite films

## Supplementary Files

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