

Mechanical and electrical properties of poly(vinyl chloride) loaded with carbon nanotubes and carbon nanopowder

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Abstract

Two groups of polymer nanocomposite samples were synthesized. One of them was Poly(vinyl chloride) (PVC) loaded with different concentrations of carbon nanopowder (CNP), while the other was PVC loaded with different concentrations of carbon nanotubes (CNTs). The dependence of the tensile mechanical parameters, rheological properties and the dc electrical conductivity on the concentration of either filler was investigated. Results revealed a lower electrical and mechanical percolation threshold of CNTs than CNP. Concentration of CNTs of 1 wt% increases the elastic modulus 2.3 times greater than CNP at a concentration of 2 wt%. The storage modulus and the complex viscosity studied at a frequency of 0.1 Hz had the same behavior as the elastic modulus. The glass transition temperature was slightly changed with the addition of either nanofiller. The behavior of the temperature dependence of the electrical conductivity was found to be temperature independent in the studied range for composites containing CNTs, while composites containing CNP showed an activated one at relatively high temperatures. At concentrations in the percolation region of either CNP or CNTs, a jump region in the I-V characteristics was observed with a highest slope of 36.35 at 1 wt% of CNTs. Otherwise the conduction mechanism of the charge carriers was determined and found to be Ohmic.

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Introduction

Carbon nanotubes (CNTs) are new materials, which have many interesting properties finding their application in different fields, such as nanoelectronic devices, automotive, aeronautic and aerospace industries.¹⁻³ Development of new materials and in particular, polymer/CNTs composites has become a new interesting area of research during the last decade. However, investigation and improvement of their physical properties are still necessary for optimization of such materials and for development of their applications. It was shown that the mechanism of electrical transfer is better in polymer/CNTs materials than in composites filled with metallic or carbon particles.⁴ A combination of conductive CNTs with an insulating polymer matrix is expected to result in electrically conductive composites at very low filler concentrations.^{5,6} It was shown that the spatial distribution of dispersed conductive filler influences the electrical conductivity of PVC and polypropylene.^{4,7} The effect of spatial distribution and geometry of CNTs on the modulus of nanocomposites using finite element analysis was studied.⁸ Results illustrated that the effects of fiber volume fraction and aspect ratio were similar to those of conventional short-fiber composites.

When the filler distribution results in the formation of a segregated structure (when the filler phase creates a conductive skeleton within the polymer matrix), the electrical conductivity is enhanced as compared to a random distribution of a filler in the polymeric matrix.⁹ For most composites, improvement of the polymer/CNTs properties strongly depends on several factors, such as orientation and distribution of CNTs in the polymeric matrix, the manufacturing process, the surface contacts and adhesion between both components.⁹

CNTs are excellent candidates for polymer matrices reinforcement because of their high strength (~ 100 times stronger than steel¹⁰) and modulus (about 1 TPa), high thermal conductivity (about twice as high as diamond), excellent electrical capacity (1000 times higher than copper), and thermal stability (2800°C in vacuum).¹⁰ Nanophased matrices based on polymers and carbon nanotubes have attracted great interest because they frequently include superior mechanical, electrical, and thermal properties. Different polymer/CNTs nanocomposites have been synthesized by incorporating CNTs into various polymer matrices, such as polyamides,¹¹ polyimides,¹²⁻¹⁴ epoxy,¹⁵ polyurethane,^{16,17} polypropylene¹⁸⁻²⁰ and poly(vinyl chloride).²¹ The mechanical properties of nanocomposites with various volume fractions of CNTs embedded in amorphous polymer matrix has been investigated.²² Results indicated that when the interaction between the CNTs and polymer is strong, the interfacial effect cannot be ignored.

In the present work the effect of both carbon nano powder and carbon nanotubes on the tensile, rheological and electrical properties will be investigated. In this

respect the stress-strain curves, elastic modulus, tensile strength and strain at break will be studied. Besides, the storage modulus, complex viscosity and loss modulus will be studied as a function of the concentration of each filler at constant frequency and room temperature will be investigated. The dynamic mechanical thermal properties at constant frequency for all composites will be studied and the glass transition temperature will be produced. Moreover, the I-V characteristics and the dc electrical conductivity as a function of fillers loadings will be studied. The conduction mechanism of the charge carriers will be determined. The dependence of the dc conductivity on temperature will be also investigated.

Experimental

Materials and preparation

A commercial grade of Polyvinyl chloride (PVC) was supplied from Sabic Company, Saudi Arabia. It was in the powder form (powder fraction of 90–120 μm , average size of 100 μm , density $\rho_p = 1.37 \text{ g/cm}^3$) was used as a polymeric matrix for preparation of composites.

Carbon nanopowder, average particle size diameter less than 50 nm and specific gravity of 1.8 was supplied by Aldrich. Carbon nanotubes with density of 2.045 g/cm^3 were supplied from the Center of Excellence in Nanotechnology, KFUPM, Saudi Arabia. Scanning Electron Microscopy (SEM) image of the CNTs is shown in Figure 1a with diameters ranging from 12.7 to 24.4 nm. The Energy dispersive X-Ray (EDX) microanalysis shown in Figure 1b satisfies the purity of used CNTs.

Initially, a desired amount of either CNTs or CNP was dispersed in Tetrahydrofuran, THF solution. This solution was then sonicated for 30 min to obtain dispersive nanofiller suspension. The desired amount of PVC was also dissolved in THF. The weight ratio of CNTs or CNP in PVC was determined as follows. A certain weights, w_p , of PVC and w_c of filler were chosen such that the total weight was $w = w_p + w_c = \text{constant}$. Then, the required ratio, w_c/w_p , was determined by increasing w_c and decreasing w_p as illustrated in Table 1. Both solutions were mixed together using a magnetic stirrer for 24 h to form nanofiller/PVC solution mixture. The final mixture was further sonicated to give a black-colored stable solution with no detectable solid precipitation. The resulting solution was poured to Petri dishes with fixed area and dried at room temperature. A series of composite films were prepared at different weight ratios (0, 0.5, 1, 2, 5, 10 and 15 wt%) of CNTs/PVC and (0, 1, 2, 5, 10 and 15 wt%) CNP/PVC films. The film weight has to be very close to the fixed weight w to ensure the correct wt% of fillers. To ensure a further interactions between the nanofillers and the PVC matrix, each prepared film was placed in a hot die at (160°C) and pressed (hot compaction) for 5 min under a pressure of 2 MPa. Finally, the die was cooled in air down to room temperature.

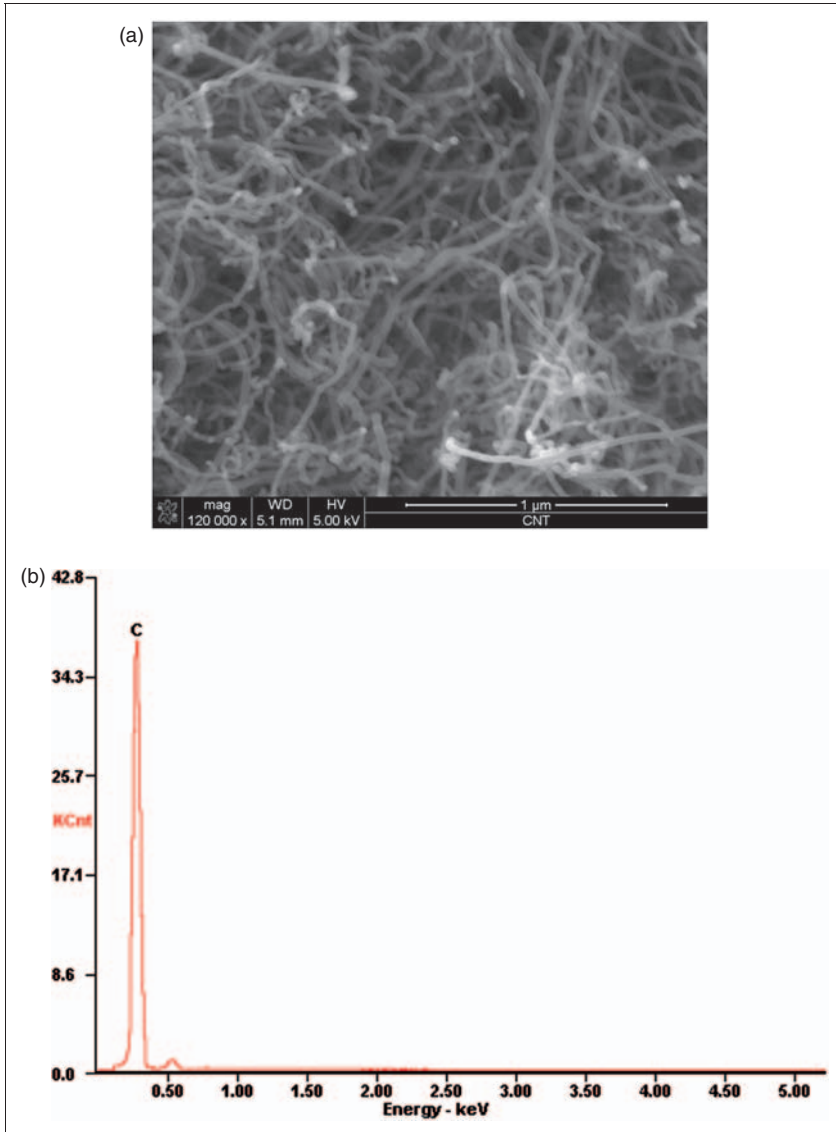


Figure 1. (a) SEM micrograph of as received CNTs and (b) EDX microanalysis of as received CNTs.

Tensile tests

The tensile tests were carried out on dumbbell-shaped specimens. The measurements were done at 25°C on an Instron 3345J8621 (Norwood) tensile machine with a grip separation of 40 mm at a crosshead speed of 5 mm/min according to ASTM D 412.

Table 1. Mix formation of CNP or CNTs/PVC nanocomposites.

Weight Ratio (wt%)	Mass of PVC (mgm)	Mass of filler (mgm)
0.0	500	0.0
0.5	497.5	2.5
1.0	495	5.0
2.0	490	10
5.0	476	24
10.0	455	45
15.0	435	65

Rheological and DMTA test

These tests were carried out on a Dynamic Mechanical Analyzer DMA Q800 (TA Instruments LLC, Delaware, USA) instrument with film clamps in dry mode.

Rheological properties measurements were done through an isothermal process at 25°C and at different frequencies ranging from 0.01 to 200 Hz. A static pre-load force (0.01 N) was applied to the sample prior the dynamic oscillating force to prevent film buckling.

For the DMTA measurements a slow heating rate of 1°C/min was employed throughout to ensure that the sample was in thermal equilibrium with the instrument. The oscillating frequency was fixed at 5 Hz.

During all measurements, the instrument was programmed to maintain the static load at 125% of the force required to oscillate the sample. It is important that the film remained in its linear viscoelastic region during measurement (to ensure that the properties observed were independent of the deformation applied and truly reflected molecular motions), and so experiments were recorded maintaining constant strain. Generally, for thin polymer films, linear viscoelastic behavior can be assured with a strain less than 0.1%, and so this limit was used.

Electrical conductivity measurements

The DC electrical specific conductivity σ_{dc} was measured using a two-electrode method. Sample was painted from both sides with a high quality silver paste and placed between two brass electrodes to ensure a perfect composite-electrode contact. The values of σ_{dc} were calculated using the equation:

$$\sigma_{dc} = \frac{h}{VS}I \quad (1)$$

where I is the electrical current measured experimentally using a digital electrometer model 6517 type Keithley, 5 kV DC power supply, coaxial cable, metal shielding, and a common ground loop, V is the applied voltage, h and S are the sample thickness and area, respectively.

Structure CNTs

The structure of CNTs was examined with an FEI NNL200 SEM (KACST, Saudi Arabia).

Results and discussions

Stress-Strain curves

The stress strain curves of PVC loaded with different concentrations of CNP and CNTs are illustrated in Figures 2 and 3 respectively. It is observed for all the plots that, at low strain, there exists a so-called elastic energy in the materials. This energy is stored in the chemical bonds in the form of strain energy prior to break. For greater strain, the stored energy is called plastic energy and it is used to describe the amount of energy residually stored in the material as internal and free (orientation) energy after break. Finally rupture of the material takes place when unstable microcracks begin to appear at the weakest sites. Once the crack exists, fracture occurs when the energy stored in the part above the crack surpasses

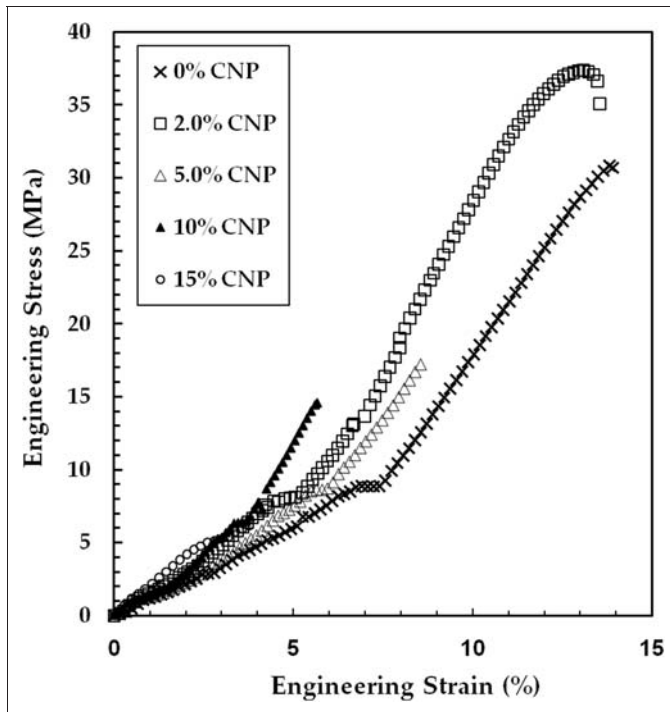


Figure 2. Stress-strain curves of PVC filled with different concentrations of CNP at a strain rate of 5 mm/min.

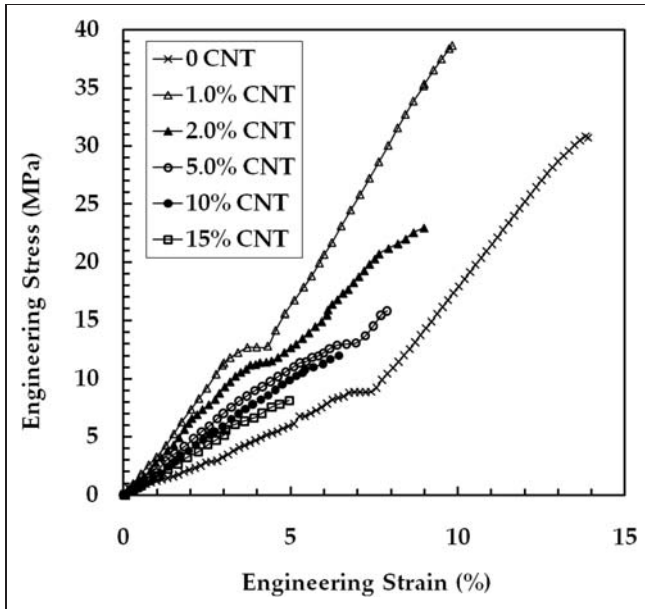


Figure 3. Stress-strain curves of PVC filled with different concentrations of CNTs at a strain rate of 5 mm/min.

the energy stored for chemical bond strain, together with the energy stored for molecular orientation around the microcrack.²³

Young's modulus, E is calculated from separate plots (not mentioned here) between $d\sigma/d\varepsilon$ versus ε for all studied composites. The plots show strain independent regions at very low strain values. These constant values are taken as Young's moduli E , at different concentrations of both CNP and CNTs which are shown in Figure (4). One weight percent of CNTs increases Young's modulus by about 200% while 2wt% from CNP increases it only by about 37%. Polymer chains may get folded around the length of the CNTs making additional entanglements which results in strengthen the composite. Besides, this may be due to direct bonding between chain ends and CNTs.²⁴ The increase in Young's modulus with adding CNP may refer to the folding of polymer chains around the powder aggregates. Entanglements between polymer chains and CNTs are larger than those for CNP which may be the reason behind the high values of E for composites filled with CNTs over those with CNP. As the filler concentration increases beyond 1wt% of CNTs and 2wt% of CNP, filler-filler contacts and so fewer contacts between fillers and polymer chains resulting in a decrease of E .

The addition of both fillers to PVC increases the tensile strength from 31 to 39 MPa at 1wt% of CNTs and from 31 to 35 at 2wt% of CNP followed by a decrease for both of them as shown in Figure 5. The observed increase at low filler content could be attributed to the interaction between filler surfaces and polymer chains endings. At higher contents of either filler, there are less filler-polymer contacts

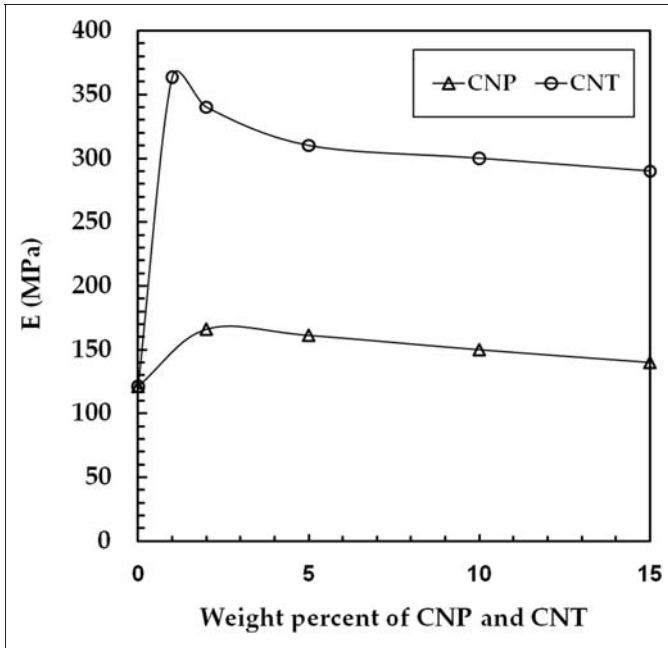


Figure 4. The elastic modulus of PVC filled with different concentrations of both CNP and CNTs.

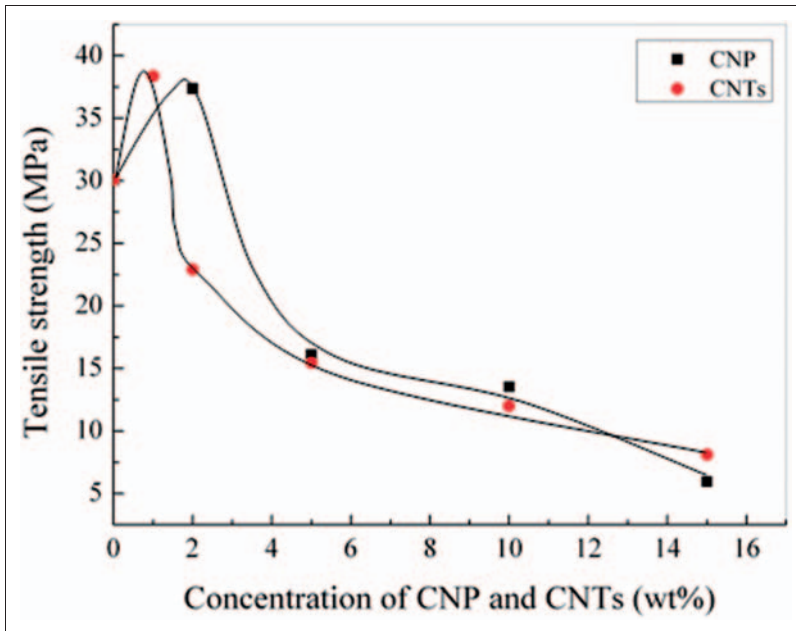


Figure 5. The tensile strength of PVC filled with different concentrations of CNP and CNTs.

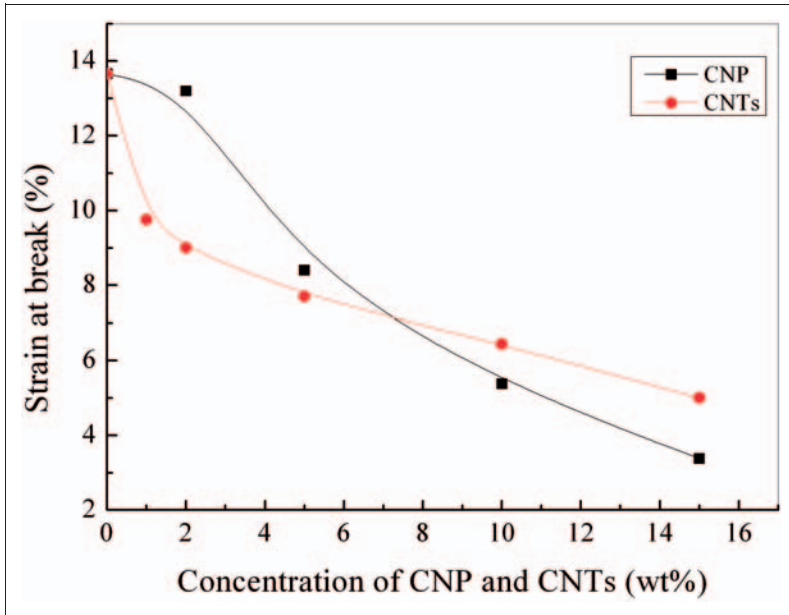


Figure 6. The strain at break of PVC filled with different concentrations of CNP and CNTs.

and consequently less filler polymer interactions. Figure 6 shows that the strain at break falls sharply when PVC is loaded with 1 wt% of CNTs followed by a gradual decrease for higher loadings. However, a small change in the failure strain is observed when PVC is loaded with 2 wt% of CNP. Behind this concentration the strain at break decreases gradually too. High tensile stresses tend to reorient, uncoil and align polymer chains along the stress direction. The presence of filler acts as obstacle to chains reorientation and prevents them to uncoil which results in a decrease in the strain at break. Higher interactions between CNTs, and polymer chains than CNP, make them highly restrict chains mobility and thus decrease the strain at break.

In general, the mechanical strength of PVC is found to improve with the addition of carbon as a nanocomposite filler. The nanosized carbon particles or tubes lodge themselves at the interface between the phases and enhance the stress transfer, which results in enhancement of mechanical properties, particularly the Young's modulus.²⁵ The polymer chains become stiffened and rigid, making the nanocomposite polymer less susceptible to stretching.^{26,27}

Rheological properties

The complex viscosity, η^* , for PVC loaded with different concentrations of CNP and CNTs measured at a driving frequency of 0.1 Hz and at room temperature is shown in Figure 7. It can be seen that η^* increases with increasing the concentration of CNTs to 1 wt% and CNP to 2 wt%. This may be attributed to the increase in physical

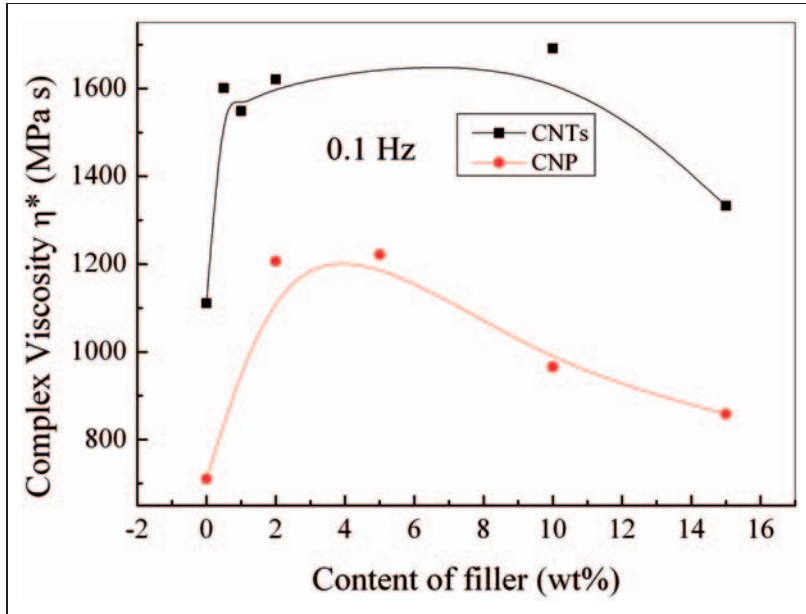


Figure 7. The complex viscosity of PVC filled with different concentrations of CNP and CNTs.

interactions between the PVC matrix and the nanofiller with high aspect ratio and large surface area, respectively. Beyond these concentrations up to 10 wt% of CNTs η^* is seen to be approximately constant which indicates no more interactions between CNTs and PVC chains. At concentrations greater than 5 wt% of CNP and 10 wt% of CNTs the complex viscosity decreases as a result of less bonding between either filler and polymer chains. The complex viscosity has higher values for CNTs than CNP, at the studied frequency, because of the high aspect ratio present in CNTs.

Figure 8 presents the dependence of the storage modulus, G' , on the concentrations of CNP and CNTs loaded PVC at a frequency of 0.1 Hz and at room temperature. Concentration of 1 wt% of CNTs increases the storage modulus of PVC matrix to approximately double its original value and then remains constant. This is in consistence with the values obtained for both the elastic modulus (Figure 4) and the complex viscosity (Figure 7). However, loading CNP up to 5 wt% does not change G' but it decreases at higher concentrations. The behavior of G' for both fillers can be explained more when we analyze the dependence of the loss modulus, G'' , on the concentration of fillers which is shown in Figure 9. Low concentrations of CNTs make interacted CNTs and polymer chains are reoriented in a new configuration. This new configuration may make polymer chains to oscillate with less phase lag with the oscillating driving frequency which results as a reduction in the loss and consequently increases the storage modulus as well as the elastic modulus, since the oscillating frequency is low (0.1 Hz) that resembles static case. On the other hand, fewer interactions between CNP and polymer chains make more

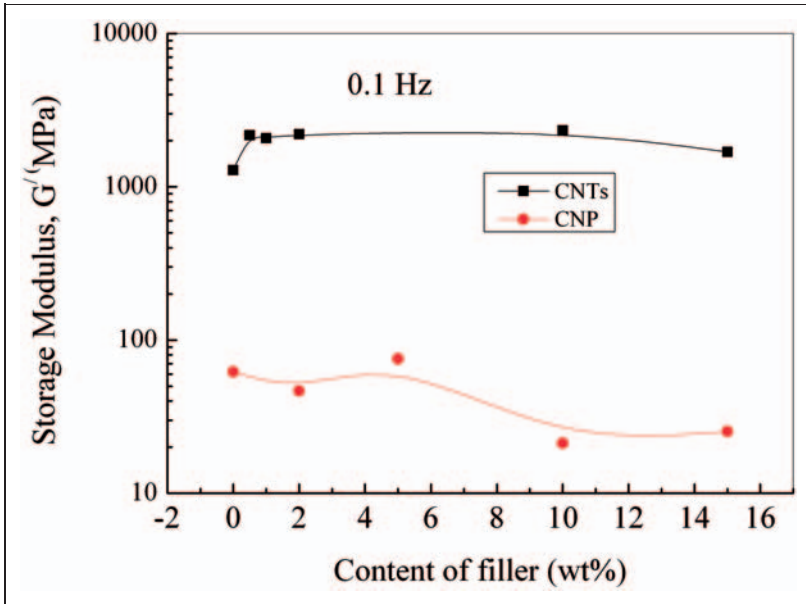


Figure 8. The storage modulus of PVC filled with different concentrations of CNP and CNTs.

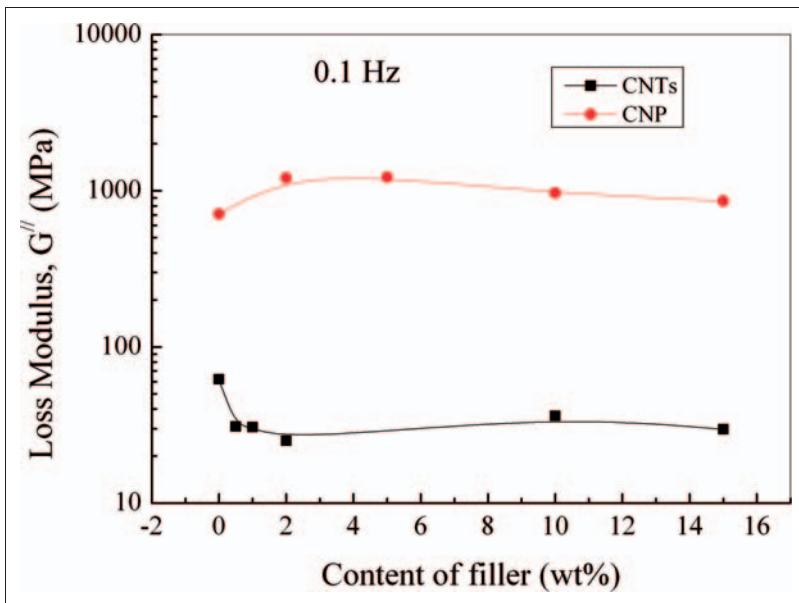


Figure 9. The loss modulus of PVC filled with different concentrations of CNP and CNTs.

frictions and more phase lag between PVC chains oscillations and the applied frequency. Besides, further frictions may take place by the un-interacted CNP particles. These appear as an increase in an energy loss or increase in the loss modulus.

Dynamic mechanical thermal properties

Figures 10 and 11 plots the storage modulus G' and the loss factor $\tan\delta$ for PVC/CNP and PVC/CNTs respectively, obtained by DMTA measurements, as a function of temperature at an oscillating frequency of 5 Hz and under a dynamic force of 1 N. A single mechanical transition has been observed in the temperature range studied and is recognized as the α -relaxation or the glass transition peak. The α -relaxation is related to the Brownian motion of the main chain associated with the glass transition and the relaxation of segments associated with it. Onset of glass transition is marked by a sharp decrease in its storage modulus, as shown in Figures 10 and 11.

The glass transition temperature of a polymer is usually taken from the peak position of loss modulus or $\tan\delta$ versus temperature curves. Peaks of $\tan\delta$ are typically found at somewhat higher temperatures depending on the intensity and/or width of the transition. Adding either filler to the PVC matrix slightly changes the $\tan\delta$ peak values and so T_g values as shown in Table 2, implying an interaction between fillers and the PVC molecules. Increasing the filler content also broadens the $\tan\delta$ peak, and lowers its intensity. The breadth of the $\tan\delta$ peak

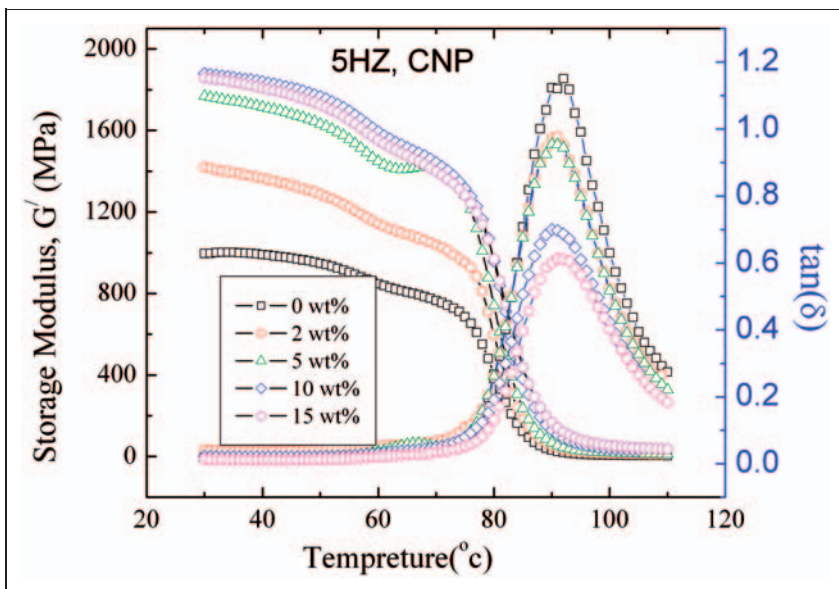


Figure 10. The storage modulus and loss tangent of PVC filled with different concentrations of CNP and CNTs at 5 Hz.

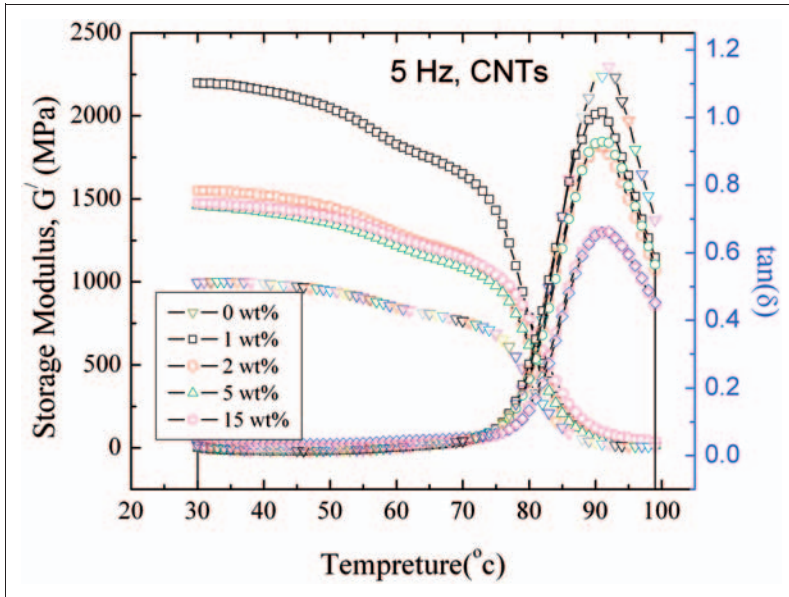


Figure 11. The storage modulus and loss tangent of PVC filled with different concentrations of CNP and CNTs at 5 Hz.

Table 2. The predicted values of the glass transition temperature at different frequencies for both CNTs and CNP.

Sample	T_g of CNTs ($^{\circ}$ C)			Sample	T_g of CNP ($^{\circ}$ C)		
	1 Hz	5 Hz	50 Hz		1 Hz	5 Hz	50 Hz
0 wt%	89.00	92.01	97.12	0 wt%	89.00	92.01	97.12
2 wt%	87.00	90.00	96.11	2 wt%	86.01	91.01	97.12
5 wt%	89.00	91.00	97.11	5 wt%	86.00	90.00	97.11
10 wt%	91.00	92.00	97.10	10 wt%	87.96	90.00	96.12
15 wt%	88.03	90.00	97.07	15 wt%	87.00	91.00	97.11

relates to the relaxation of the polymer chain. The interaction between the fillers surfaces and the PVC molecules, and the constraints on the mobility of the polymer chains due to their being tethered to the inorganic nanofiller widen the range of temperatures covered by the $\tan\delta$ peak. Hence, the breadth of the $\tan\delta$ peak increases with the filler content. The height of the $\tan\delta$ peak measures the energy-damping characteristics of a material. Fillers are well dispersed in the polymer matrix, so they directly enhance the stiffness of the composites. The height of the $\tan\delta$ peak thus declines as fillers content increases.

Current–voltage characteristics

The current–voltage characteristics of PVC loaded with different concentrations of CNP and CNTs are shown in Figures 12 and 13, respectively. For concentrations 0, 5, 10 and 15 wt% of CNP and 0, 10 and 15 wt% of CNTs the curves show straight lines between $\log(V)$ and $\log(I)$ with slopes of 1.08, 2.24, 1.05 and 1.10 for CNP and 1.08, 0.94 and 1.13 for CNTs, respectively. This indicates an Ohmic conduction mechanism for samples containing 0, 10 and 15 wt% of both types of fillers. A non linear behavior with three distinct regions appears for concentrations of 2 wt% of CNP and 1, 2 and 5 wt% of CNTs composites. The charge conduction in polymeric materials consists of the charge injection from the electrode into the material and the charge transport inside the polymeric material itself. In general, the charge injection mechanism is reasonable when the whole charge conduction is mostly influenced by the charge injection, whereas the charge transport mechanism is suitable when the effect of the injection barrier is negligible compared with the bulk resistance of the material. The former and the latter are called the injection-limited and the bulk-limited conduction, respectively.

The charge transport mechanism based on the power law model $I \approx V^m$ has been widely used for the analysis of the conduction phenomenon in organic or polymeric materials.^{28–30} The model is divided into three regimes, Ohmic ($m = 1$), trap-free space charge limited ($m = 2$), trap charge limited ($m > 2$),³¹ where m is directly related to trap state; and named the non-linear coefficient. As the values of

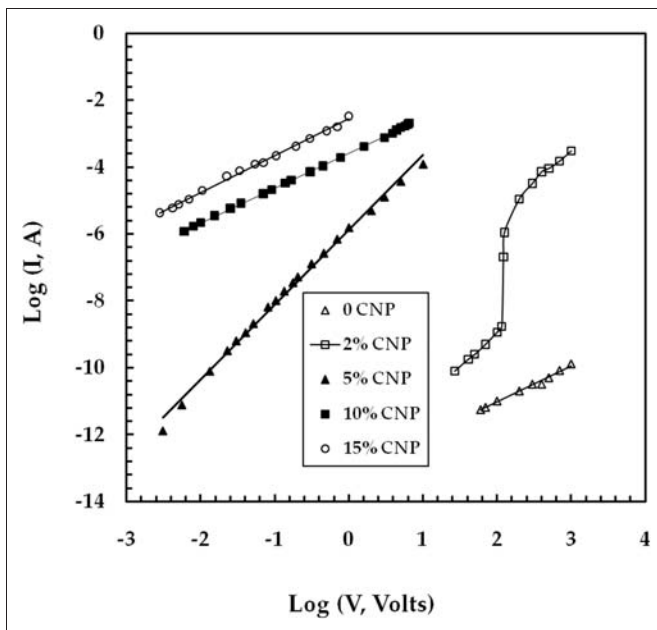


Figure 12. $\log(I)$ - $\log(V)$ characteristics of PVC filled with different concentrations of CNP.

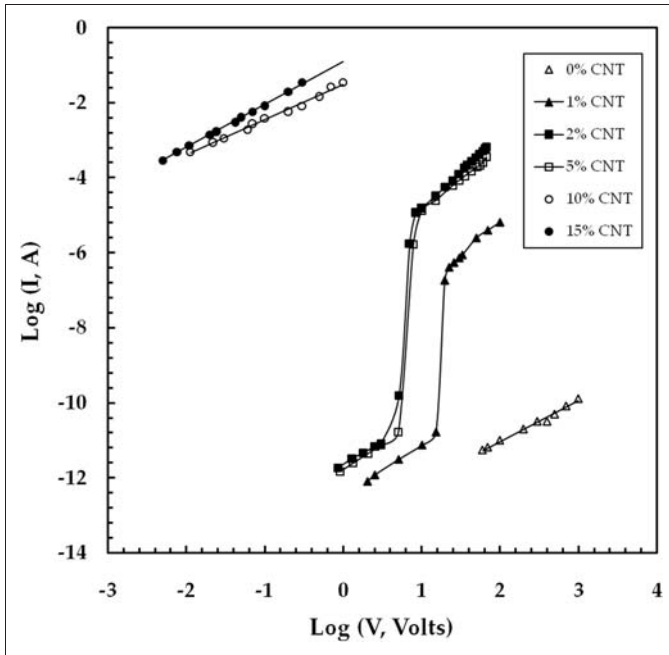


Figure 13. Log(I)-Log(V) characteristics of PVC filled with different concentrations of CNTs.

m increases the traps becomes larger or deeper. The calculated values of m (= slope of $\text{Log}(V)$ versus $\text{Log}(I)$) are 5.6 for the concentration 2 wt% of CNP and 36.4, 13.6 and 25.9 for concentrations 1, 2 and 5 wt% of CNTs, respectively.

Dc electrical conductivity

Polymers and plastics are insulators to which conductivity is imparted by addition of finely divided or colloidal fillers of high intrinsic conductivity, such as carbon black, CNP and recently CNTs. At low loading of either CNP or CNTs in PVC, the conductivity of the composite is essentially equal that of the polymer. As the loading is increased, a percolation threshold or critical loading is reached where the conductivity starts to increase rapidly as shown in Figure 14 for PVC loaded with CNP and CNTs as a function of filler loading. The figure can be divided into three distinct regions. Region A, which starts from concentration 0 phr up to approximately 0.5 phr for CNTs and 1 phr for CNP. This region is characterized by low conductivity. The fillers are dispersed inside the polymer matrix and there are no paths for charge carriers. This region is named dispersed region. The region where conductivity increases rapidly (region B) is called the percolation region. In this region, conductivity is limited by barriers to pass the charge carriers from one filler conducting site to another which is close but

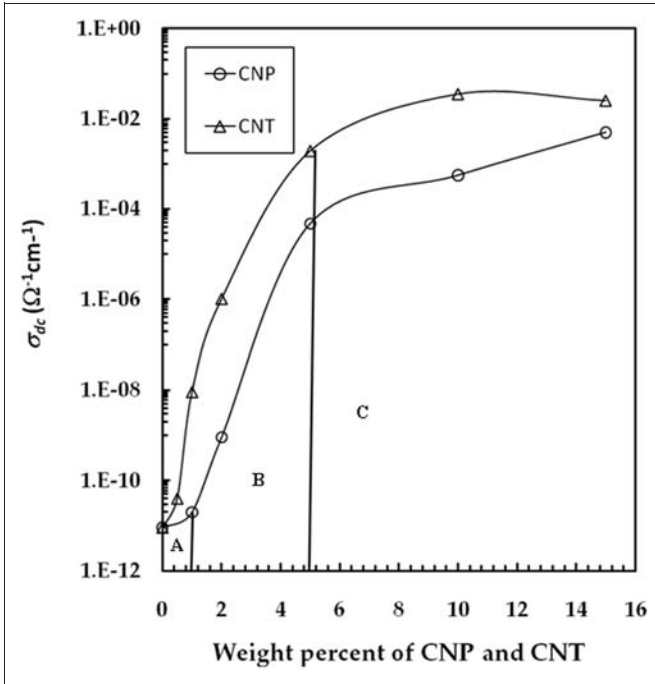


Figure 14. Dependence of the dc electrical conductivity of PVC filled with different concentrations of both CNP and CNTs at room temperature.

not in contact. In the last region C, the fillers are in contacts and the conductivity of the matrix comes mainly from that of the filler and it is called attached region. As a result of the high aspect ratio of CNTs over CNP, one can notice that the percolation concentration threshold for CNTs is less than that for CNP. Besides, CNTs make shorter paths for charge carriers than CNP. This seems to be the reason behind the high values of conductivity of CNTs over CNP at same concentrations.

Figures 15 and 16 illustrate the temperature dependence of the PVC polymer loaded with different concentrations of CNP and CNTs, respectively. The electrical conductivity at 293 K is found to increase with the addition of either CNP or CNTs as discussed before. The $\sigma - 1000/T$ curves at low filler loadings can be divided into two regions for both fillers. The first region lies above room temperature at which the behavior of conductivity is temperature independent. This behavior may be attributed to the fact that, the relative separation distances between fillers with respect to PVC are unchanged with heating. This behavior is observed for the whole studied temperature range for samples containing 2 and 5 wt% of CNTs. At higher temperatures the whole samples except those containing 10 and 15 wt% of CNTs, have activated behavior with increasing temperature. In this range of temperature, charge carriers are activated and jump the barriers between filler

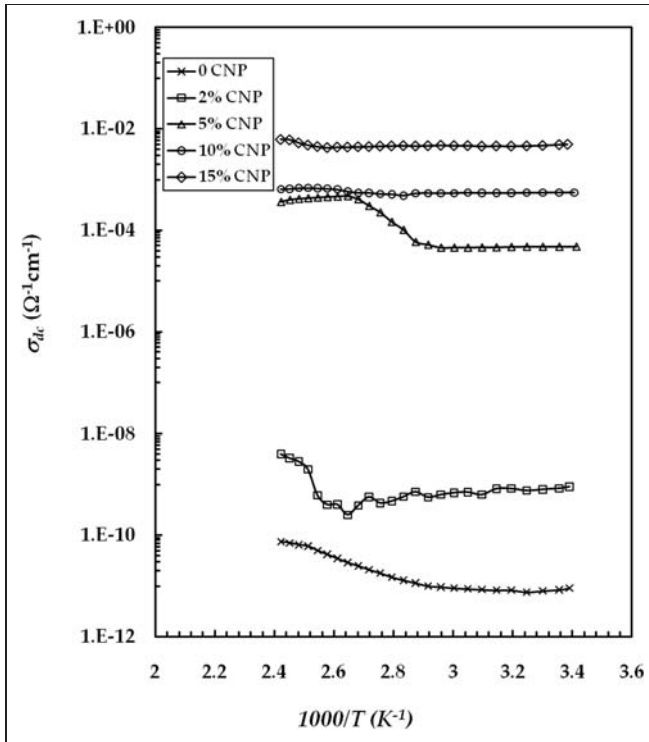


Figure 15. Temperature dependence of the dc electrical conductivity of PVC filled with different concentrations of CNP.

aggregates. This appears as an increase in conductivity. This behavior is found to obey the well known Arrhenius relation:

$$\sigma_{dc} = \sigma_{odc} \exp[-E_g/k(T - T_o)] \quad (2)$$

where σ_{odc} is the electrical conductivity at temperature T_o , σ_{dc} is the electrical conductivity at temperature T , k is Boltzmann constant and E_g is the activation energy with values shown in Table 3. The Table shows highest value of activation energy of 0.79 eV at 5 wt% loading of CNP. At concentrations of CNP greater than 5 wt%, E_g decreases because of barrier height limitation between conducting sites in the polymer matrix. The presence of CNTs with very low concentrations appears in a decrease of this height which results as a decrease in the values of the activation energy. At concentrations greater than 2 wt% of CNTs, the activated behavior of the electrical conductivity with temperature disappears because of the attached configuration of CNTs. The advantage of CNTs over CNP is that, the behavior of $\sigma - 1000/T$ curves at concentrations above 2 wt% of CNTs is approximately

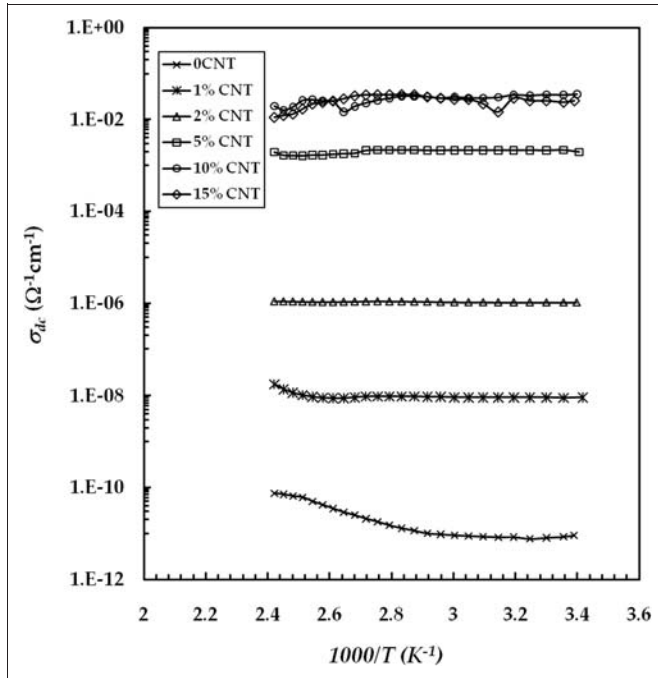


Figure 16. Temperature dependence of the dc electrical conductivity of PVC filled with different concentrations of CNTs.

Table 3. The calculated values of the activation energy for PVC filled with different concentrations CNP and CNTs.

Concentration of filler (wt%)	E (eV) = CNP	E (eV) CNTs
0	0.38	0.38
1	–	0.31
2	0.64	0.019
5	0.79	–
10	0.11	–
15	0.23	–

temperature independent. Thus, it can be better employed for electronic and microwave devices working at different temperatures.

Conclusions

Study of the mechanical properties of PVC/CNTs and PVC/CNP composites depending on the content of nanofillers demonstrate an increase in Young's

modulus by 2.3 times at concentration of 1 wt% of CNTs and 1.4 times at 2 wt% of CNP. The tensile strength initially increases up to concentrations of 1 wt% and 2 wt% of CNTs and CNP respectively, and then decreases with increasing either nanofiller. However, the strain at break is found to decrease with increasing either filler in PVC host. The behavior of the storage modulus and the complex viscosity at a frequency of 0.1 Hz and at room temperature is similar to that of the Elastic modulus for PVC/CNTs composites, while the storage modulus is different for PVC/CNP. The addition of either filler to PVC slightly changes the glass transition temperature.

The presence of nanofillers reduces the percolation concentration threshold from 1 wt% CNP to 0.5 wt% CNTs. It is caused by high anisotropy of the CNTs and CNP and the presence of a segregated structure of them within the polymer matrix with distribution of nanofillers on the boundaries between the polymer grains.

Composites containing CNTs can be better employed for electronic and microwave devices working at different temperatures since their electrical conductivity is independent on temperature in the studied range. Concentrations of either nanofiller in the percolation region produce a jump region in the I-V curve. The conduction mechanism is Ohmic for other composites.

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