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Dielectric Relaxation and Thermal Stability of Epoxy/Polyurethane - Carbon Nanofiber Composites

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ABSTRACT

The main purpose of this study is to improve the dielectric relaxation properties and thermal stability of epoxy/polyurethane blend by dispersing carbon nanofibers, the EP/PU-CNF's was fabricated by dispersing a weight fraction of (10%) of the CNF's into EP/PU blend. The data of AC conductivity have been analyzed in the light of the correlated barrier hopping model (CBH) and percolation theorem. The dielectric measurements were carried out for all samples over the frequency range of (102-107) Hz and over temperature range of (293-463) K0. It is found that all samples displayed a wide dielectric dispersion, thus the values for dielectric constant and dissipation factor give a direct evidence of Debye relaxation existence which leaves a wide distribution of relaxation time. Eyring's relaxation rate equation has been used to determine the thermodynamic parameters (Gibbs free energy of activations (ΔG), and entropy (ΔS)). The results (ΔG), and (ΔS) reveal a strong intermolecular interaction in all tested samples. The glass transition temperature (T_g) values were exhibited by differential thermal analysis (DTA) and estimated by dielectric measurements (DM). The thermal stability of EP/PU blend and EP/PU-CNF's composite was evaluated by thermal gravimetric analysis (TG) and deferential thermal gravimetric analysis (DTG), the results showed that the carbon nanofibers improved the thermal stability of the composite, hence the char yield at a temperature greater than (700) C0 reached to (31.08) %, while it is only (8.4) % for the EP/PU- blend.

Keywords: Carbon nanofiber, Dissipation factor, Epoxy based nanocomposite, and Thermal stability.

INTRODUCTION:

Carbon nanofibers possess characteristics that distinguish them from other materials. These properties include thermal, mechanical, and electrical attributes. For example, CNFs may exhibit the high thermal conductivity, excellent mechanical strength, and superior electrical conductivity compared to other materials. The remarkable properties of CNFs have garnered significant attention from the scientific community. Researchers are interested in exploring

and harnessing these properties for various applications across different fields (Rahmat M. *et al.*, 2011; Kenry *et al.*, 2017) due to this it has been widely used to reinforce polymeric materials to improve their mechanical properties (high flexibility and light weight) and their thermal and electrical properties in an effort to achieve desirable properties such as high dielectric constant (Han C. *et al.*, 2010; Shettar M. *et al.*, 2020; Rathinavel S. *et al.*, 2021; Kooti M. *et al.*, 2021). The research into polymer/carbon nanofiber

(CNF) nanocomposites has been extensive, particularly focusing on their dielectric properties and thermal stability and much emphasis has been placed on improving the dielectric properties by obtaining high dielectric constant and relatively low electrical loss (Jomaa M. *et al.*, 2015; Begum S. *et al.*, 2018). The epoxy resin is most often used as the polymer matrix due to its series of interesting properties like high specific strength, good stiffness, chemical resistance, dimensional stability and also strong adhesion to the embedded reinforcement (Chang J. *et al.*, 2012; Zhang X. *et al.*, 2012). Polyurethane coatings indeed offer several advantageous properties such as high hardness, wear resistance, radiation resistance, and peel strength, which make them suitable for various applications. However, it's true that they may have limitations in terms of corrosion resistance and temperature resistance when compared to some other coating materials (Jianxin Zhang *et al.*, 2022). Dielectric properties and thermal stability can be significantly improved by mixing epoxy and polyurethane resins with the addition of carbon nanofibers (CNF's) because of their ability to improve these properties (Ameli A. *et al.*, 2014; Yang X *et al.*, 2014). In light of the results of previous studies, the main objective of this research is to study the dielectric relaxation and thermal stability of epoxy/ polyurethane blends and their composites reinforced with carbon nanofibers (CNF's). During this experimental study, AC conductivity was examined as a function of temperature, and over a frequency range (10^2 - 10^7) Hz. The real and imaginary parts of the dielectric constant and the dissipation factor were also measured as a function of temperature and

frequency. Furthermore, all blends and their composites samples were scanned using DTA and TG to study the thermal stability of the blends and their composites. The composites fabricated in this study showed good dielectric properties and high thermal stability, which makes them useful materials in many manufacturing and electronic applications.

METHODOLOGY:

The materials listed below were used according to the mention characteristics described in this study.

a) Epoxy Resins (DGEBA, YD-128).

Description: low molecular weight liquids with an epoxide equivalent weight of 176 G, supplied by Kukdo Chem. Co., Korea.

b) Curing agent pathalic anhydride (PA).

Description: Phthalic anhydride is commonly used as a curing agent for epoxy resins, supplied by Kukdo Chem. Co., Korea.

c) Polyurethane (PU).

Description: A commercial resin type (TEK-CAST), supplied by TEKCAST- Industries, New Rochelle, NY 10801, USA

d) Curator Isocyanides (HYG)

Description: Isocyanides are likely used as curing agents for the polyurethane resins, supplied by TEKCAST- Industries, New Rochelle, NY 10801, USA

e) The Carbon nanofibers (CNF's)

Description: purity 99.9, Diameter $1\mu\text{m}$ and length less $100\mu\text{m}$ supplied by Nanoshell company, India was used in this study.

Table 1: Carbon nanofibers (CNF's) specifications.

Diameter	Length	Purity	Molecular weight	Density	Melting point	Color
$<1\mu\text{m}$	$<100\mu\text{m}$	99.9	12.01g/mole	2.1g/cm ³	3652-3697 C ⁰	Black

Samples Preparation

An open-mold casting technique was used to perform the epoxy/polyurethane blends and their composites in this study. Epoxy and polyurethane blends were prepared in this study with different weight fractions (90%-10%), (80%-20%), and (70%-30%), where the blend (80%-20%) was chosen to prepare the composite by adding a fixed weight fraction (10%) of carbon nanofiber of Diameter $<1\mu\text{m}$ and length less $<100\mu\text{m}$.

Measurements

Specimen Holder

The specimens of the epoxy/polyurethane blends and their composites, is fixed in a specimen holder. This holder ensures that the sample remains in place during testing.

Temperature Controller Oven

The temperature-controlled oven, manufactured by Heresies Electronic, is used to subject the specimen to

controlled temperature conditions. This allows for testing the specimen's electrical properties at different temperatures.

Dielectric Analyzer

The dielectric analyzer used in the experiment is of Hewlett Packard model, specifically HP4274A and HP4275A. These analyzers are designed to measure various electrical properties of materials, including dielectric parameters.

Terminal connections and measurements

The specimen holder has high and low terminals, which are connected to the dielectric analyzer for electrical measurements. The third grade is connected to the earth, for grounding purposes to prevent electrical interference. Three dielectric parameters were measured directly from above setup total resistance (R_T), total capacitance (C_T) and dissipation factor $\tan(\delta)$. Thermal stability was performed by thermogravimetric analysis (TG) and differential thermal analysis (DTA) (NETZSCHSTA 409PG/PC) the measurement performed under heating rate of 20 C⁰/min over the range (0-800) C⁰.

RESULTS AND DISCUSSION:

A.C Conductivity ($\sigma_{ac}(\omega)$)

Fig. 1 shows the variation of AC conductivity ($\sigma(\omega)$) as a function of frequency (ω) at various temperatures

(T/K⁰) for A) EP8%/PU20%B) EP/PU-CNF's have revealed that AC conductivity ($\sigma(\omega)$) increased with increasing frequency and temperature for all tested samples. Hence, there are two factors that affect $\sigma(\omega)$ for the EP80%/PU20% mixture, namely the mobility of the ions and the mobility of the polymer backbone (main chain). Moreover, the movement of ions contributes only at high frequencies and temperatures (T. Jeevanada. Siddaramaiah, 2003). Therefore the increase in the $\sigma(\omega)$ at low frequencies over the range (100-400) Hz is attributed to interfacial polarization (T. Jeevanada *et al.*, 2002) while the rapid increase in $\sigma(\omega)$ with increasing frequency at frequencies greater than (10³) Hz indicates the effect of electronic polarization, and the conductivity is pure ac conductivity $\sigma(\omega)$ in this region. The composite (EP/PU-CNF's) shows a big jump in $\sigma(\omega)$, which is increased by three orders to reach (9.11×10⁻⁴ Scm⁻¹) at frequency of 1MHz. Thus the discussion is truly changed into a semiconducting case. This jump in $\sigma(\omega)$ was achieved by incorporating (10%) weight fraction of CNF's to EP/PU blends, which can be used to dissipate the static charges. This behavior occurs because all fiber molecules in the composite network are connected to each other forming tri-phase continuity, and all are contributed to the transport of charges (Maffezzoli A *et al.*, 1994).

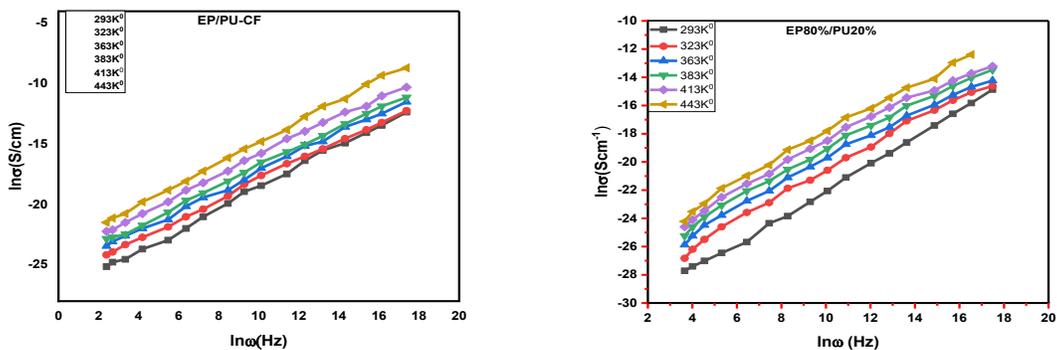


Fig. 1: Electrical conductivity of EP/PU blend and EP/PU-CNF's composites at different frequencies and temperatures.

Fig. 2 shows the variation of exponent (s) of EP/PU blend and EP/PU-CNF's composites as the function of temperature. The result reveals that the exponent values are less than unity for EP/PU blend and EP/PU-CNF's composites. Also these values are decreased

with temperature increasing. This behavior of the exponent (s) can be explain with the correlated barrier hopping model (CBH) model which proposed by Elliot (Maffezzoli A *et al.*, 1994).

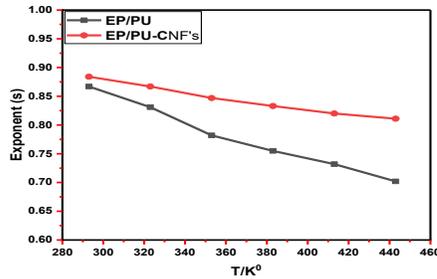


Fig. 2: The Exponent (s) of EP/PU blend and EP/PU-CNF's Composites at different temperatures.

Fig. 3 shows the variation of activation energy (E_{ac}) of (A) EP/PU blend (B) EP/PU-CNF's Composites as a function of frequency, the activation energy is estimated by Arrhenius equation. Each sample reveals three activation energies, the values of (E_{ac}) were decreased with increasing frequency, such result complies with the theory of CBH model. In light of the published literature, these results are in good agreement with published results for both epoxy blends and epoxy-based composites (Carmona F. *et al.*, 2017). The first critical temperature coincides approximately with glass transition temperature (T_g) of polymers.

This phenomenon was observed in the epoxy resin by Warfield and Petree (Warfield R.W. & Petree M.C., 1962). The change in the slope at T_g can be explained by increases in the free volume and active diffusion motions of the chain segment (Saito S. *et al.*, 1968). The free volume would also make possible great increase in the mobility of the charge carriers. The composite reinforced by carbon nanofiber show smallest values of (E_{ac}), this result supports the changing of this composite into a semiconducting phase.

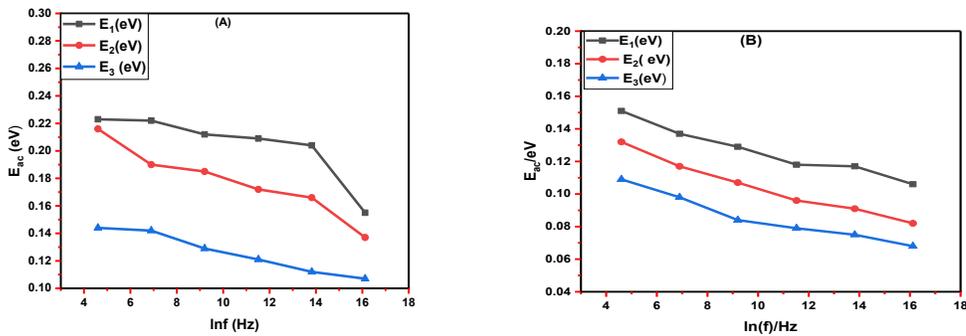


Fig. 3: The activation energy (E_{ac}) of (A) EP/PU blends (B) EP/PU-CNF's Composites at different frequencies.

Dielectric Study

Fig. 4 shows the real part of dielectric constant (ϵ_r) as a function of frequency (ω) at various temperatures for the A) EP/PU blend and B) EP/PU-CNF's composite. The results show that the values of (ϵ_r) in EP/PU blend samples decreases with increasing frequency to reach a lower values at high frequency which represent the onset frequency. This result can be explained by the fact that the electrode blocking layers is dominated mechanism at low frequency region (Maffezzoli A. *et al.*, 1994). Thus the dielectric behavior is affected by electrode polarization in the range of (10^2 - 10^4) Hz.

Likewise, the value of the real part of the dielectric constant increases slowly with increasing temperature until it reaches the highest value, this behavior was consistent with Debye- type dielectric dispersion characterized a long relaxation time (Radzuan. N. A *et al.*, 1989). **Fig. 5B** shows that the values of (ϵ_r) are affected by the adding of reinforcement material (CNF's). However; the real part of dielectric constant reveals highly frequency dependence at the frequency range over (10^2 - 10^7) Hz. This result can be explained by an intra-chain transport mechanism involving polarons is exist, this mechanism allows an explan-

ation of high dielectric constant values due to nomadic polarization which is observed in many polymer

composites (Pohl H. A. & Pollok. M., 1975).

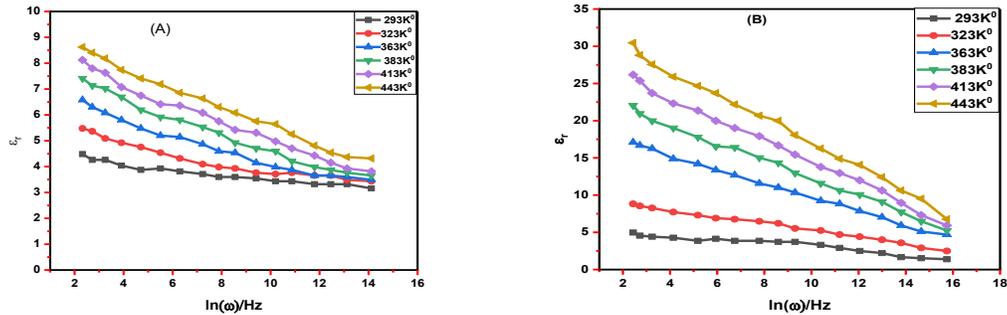


Fig. 4: The real part of the dielectric constant (ϵ_1) of (A) EP/PU blend (B) EP/PU-CNF's Composites at different frequencies and temperatures.

Fig. 5 shows the imaginary part of dielectric constant (ϵ_2) as a function of frequency (ω) at various temperatures for the (A) EP/PU blend and (B) EP/PU-CNF's composite. **Fig. 6A** show the decrease in values of (ϵ_2) with increasing the frequency further 10^3 Hz, where strong relaxation contributions prevail (Narula A. K. *et al.*, 2000). In the frequency greater than 10^3 Hz the values of (ϵ_2) are grown up and the loss peak is exist at a constant position, where the migrating charges play the major role in dielectric polarization (Butta E. *et al.*, 1995). At high frequencies the values of (ϵ_2) are decreased rapidly with the frequency this behavior is

referred to an electrode blocking layers polarization dominance (Han C. *et al.*, 2010). **Fig. 6B** shows that the values of (ϵ_2) are affected by the adding of reinforcement material (CNF's). However the result shows that the (ϵ_2) was decreased with increasing the frequency and the strong dispersion exhibited at (4×10^3) Hz the composite. At the strong dispersion region, the migrating charges play a major role and the space charge polarization is dominant mechanism, while at high frequency the relaxation contribution prevail (Ming Qin. *et al.*, 2022).

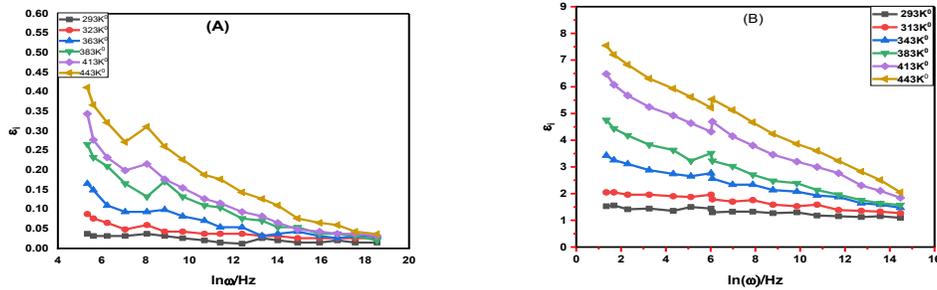


Fig. 5: The imaginary part of the dielectric constant (ϵ_2) of (A) EP/PU blend (B) EP/PU-CNF's Composites at different frequencies and temperatures.

Fig. 6 shows the variation of dissipation factor ($\tan(\delta)$) as a function of frequency at various temperatures for (A)- EP/ PU blends and (B) EP/PU-CNF's composites. **Fig. 6A** shows that the loss peak occurs at the region of strong dispersion (10×10^3) Hz. This behavior is typical of many dielectric materials, the common feature of which is presence of strong carrier polarization rising from high densities of low mobility

charge carriers (Ming Qin *et al.*, 2022). At low temperatures the $\tan(\delta)$ show weak frequency dependent, while at high temperatures greater than 353K the strong frequency dependence phenomenon is existing. This result supports the discussion present on $\sigma_{ac}(\omega)$. Also this result consistent with Debye- type dispersion, which is suggest that the Debye relaxation had being adequate to explain the dielectric data (Papa-

thanassiou A. N. *et al.*, 2007). **Fig. 6B** shows a strong dispersion occurs in EP/PU-CNF's composites, over the frequency range (10^2 - 10^3) Hz and the loss peaks does not show any significant shift with increasing

temperature. This result has been attributed to strong carrier polarization a rising from high densities of low mobility charge carriers (Ming Qin *et al.*, 2022).

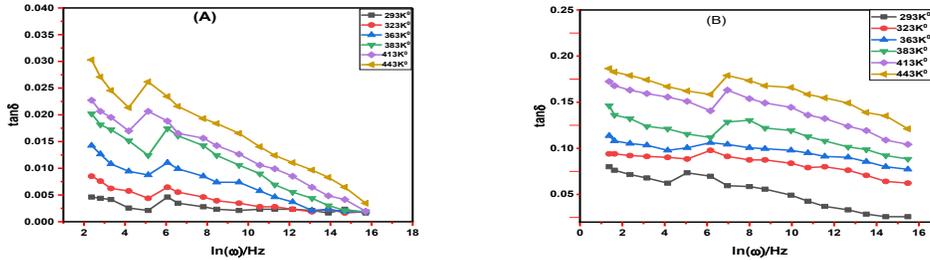


Fig. 6: The dissipation factors ($\tan \delta$) of (A) EP/PU blend (B) EP/PU-CNF's Composites at different frequencies and temperatures.

The thermodynamic parameters Gibbs free energy of activation (ΔG), and entropy (ΔS) are estimated from dielectric relaxation measurements according to Eyring's theory (Eyring H. 1936). **Fig. 7** shows the variation of thermo-dynamic parameters (ΔG), and (ΔS) as function of temperature. The results revile that the values of free energy of activation (ΔG) and the entropy (ΔS) are increased with increasing temperature. The physical reason of this result can be related to the amount of thermal energy which is absorbed by the polymer chains, which a rise the segmental motion of molecules (Alshammari A. H *et al.*, 2023). While the CNF's composite appears high values of energy of activation ΔG to reach (67KJ) at 443K0, and this result confirms a strong intermolecular interaction.

Such resultant composites possess a high degree of cross linking (Eyring H., 1936).

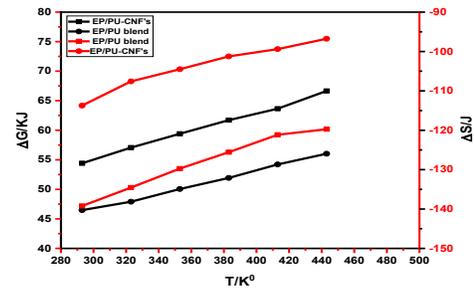


Fig. 7: The Gibbs free energy of activation (ΔG), and entropy (ΔS) EP/PU blend and EP/PU-CNF's Composites at different temperatures.

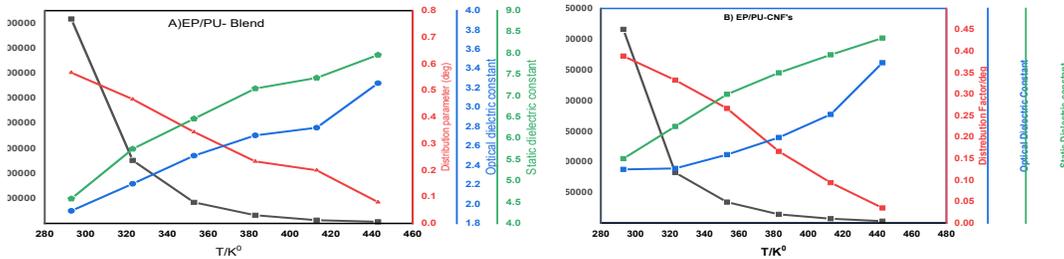


Fig. 8: The dielectric relaxation parameters ($\tau, \alpha, \epsilon_s, \epsilon_\infty$) of (A) EP/PU blend (B) EP/PU-CNF's Composites at different temperatures.

Fig. 8 shows the values of static dielectric constant (ϵ_s) and optical dielectric constant (ϵ_∞) distribution parameter (α) and relaxation time of (A) EP/PU blend (B) EP/PU-CNF's Composites at different temperatures. The values of relaxation time (τ) and distribution parameter (α) EP/ PU blends and composites are systematically decreased with increasing temperature. This result due to the orientation polarization, since this type of polarization is very sensitive to the temperature (Afiqah N. A. *et al.*, 2017). The result shows that the

meter (α) EP/ PU blends and composites are systematically decreased with increasing temperature. This result due to the orientation polarization, since this type of polarization is very sensitive to the temperature (Afiqah N. A. *et al.*, 2017). The result shows that the

values of molecular relaxation time (τ) decreased with increasing temperature. Also result shows that the static and optical dielectric constants are systematical increase with increasing temperature. This can be explained as; the temperature increases the dipoles slowly get activated and attain freedom of rotation while the dielectric remains in the solid phase (Afiqah N. A. *et al.*, 2017).

Thermal analysis and Thermal Stability

Fig. 9 shows the differential thermal analysis (DTA) thermograms of EP/PU blend and their composites. The glass transition temperature (T_g) values as exhibited by DTA and estimated by dielectric measurements (DM) are shown in **Table 2**. The results show that the blend had only one (T_g) while the composites exhibited two values of (T_g). These two values of T_g of the composites probably reflected the

fact that permanent compatibility of composites networks was impossible (Alshammari A. H. *et al.*, 2023). DTA and DM values of T_g show small variation 0.78%-1.6% for EP/PU blends this result attributed to high compatibility between two networks. The two components of the blend are cross-linked networks, hence the single T_g may be attributed to the permanent interpenetration of the polymer networks and well give arise to improve compatibility and less phase separation (Venkatesh M. *et al.*, 2023). The values of T_g for CNF's composites are strongly influenced by the adding of the dispersed phases. The composites reveal two glass transition values the first can be referred to the polymer blends and the second one may be referred to the interface between the polymers and the CNF's.

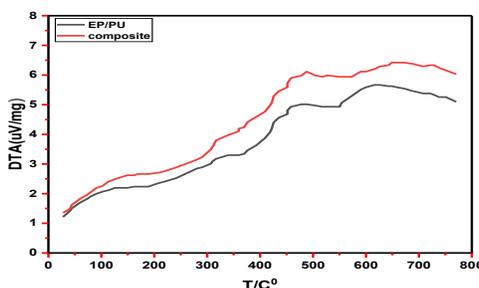


Fig. 9: DTA thermograms of EP/PU blend and EP/PU-CNF's.

Table 2: The values of glass transition temperature (T_g) of EP/PU blend and EP/PU-CNF's composite which are estimated by DTA and dielectric measurements (DM).

Specimens	T_g (DTA) /K0	T_g (DM) / K0	(T_g (DTA) - T_g (DM))%
EP/PU Blend	378	375	0.79%
EP/PU-CNF's	363,386	396	9.1%

Fig. 10A shows TGA thermograms of EP/PU blend and EP/PU-CNF's composite. **Table 3** summarizes the result of onset (initial weight losses) temperature (T_i), the $T_{50\%}$ value represents the temperature at which the sample has lost 50% of its initial weight due to decomposition, volatilization, or other thermal processes, the sample lost 80% of its initial weight called (T_f) and weight fraction of char yield at 800 °C (wt% char at 800 °C). The EP/PU blend the adding of PU resin reduces the char yield and (T_f) of the blend. This result agrees with the nature of PU which possess two steps of degradation, the first one is well correlated with the hard segment concentration; polyol, while the

second step is correlated with degradation of the soft segment; isocyanate, (Shokralla S. A. and Al Muikel. N. S., 2010). The TGA thermo-grams show that the EP/PU blend is stable up to 341°C and completely degrades around 600°C in three steps, the first step weight loss occurs over the temperature range 220-350 °C, due to the loss of bound dopant and the degradation of segment (polyol) referred to PU and EP resins [32]. The major weight loss occurs in the range 377-500°C corresponds mainly to the loss of main chain degradation include the bisphenol-A and isocyanate (A. Maffezzoli *et al.*, 1994). The

degradation takes place at temperatures above 500°C through thermo-oxidative reactions (Venkatesh M., 2023). **Fig. 10B** illustrates TGA curves of EP/PU-CNF's composites. TGA curves reveal that the composite is stable up to 200°C and then completely degraded around 600°C in three steps. The first step weight loss occurs at 220°C is due to the loss of bound dopant and the degradation of segment (polyol). The second weight loss represent the major loss is exhibited at temperature of 371°C this is due to the degradation of main chain includes the bisphenol-A and isocyanate (Venkatesh M. *et al.*, 2023). The final

step is located at temperatures above 700°C, and this loss refers to thermo-oxidative reactions (Shokralla S. A. & Al Muikel N. S., 2010; Abubakre O. K. *et al.*, 2023). **Table 3** shows that the char yield at 800°C of the blend and its composite are strongly affected by the reinforcement phases. Thus the reinforcement materials enhance the char yield. Since the rate of degradation of the composite reduces by adding the reinforcement materials. The thermal stability enhances by reinforcement materials at high temperature region, T_f increases rapidly to equal 711.6°C.

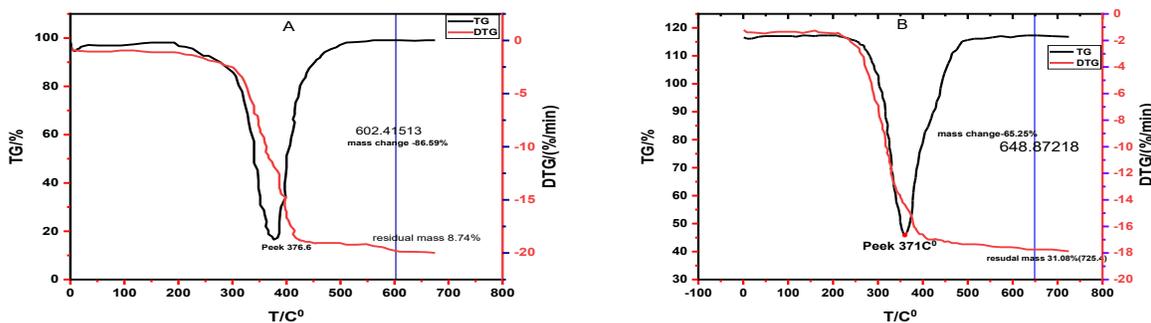


Fig. 10: TGA thermograms of A- EP/PU Blend B- EP/PU-CNF's Composite.

Table 3: The experimental data of thermal stability (T_i , $T_{50\%}$, T_f , and wt% char at 800°C).

Sample	Thermal Stability Parameters			
	T_i/C^0	$T_{50\%/C^0}$	T_f/C^0	wt% char at 800°C
EP/PU Blend	348.5	392.1	440.5	8.74
EP/PU-CNF's Composite	332.9	371	711.5	31.08

CONCLUSION:

Based on the information provided about the study involving the EP/Polyurethane blend and EP/ Polyurethane -CNF's composites, several conclusions and observations can be drawn:

- The addition of dispersive phases such as polyurethane resin type novolac and embedding CNF's into the EP resin leads to improvements in various electrical properties.
- The dielectric properties of the materials, including parameters like distribution parameter (α), macroscopic relaxation time (τ_0), and static dielectric constant (ϵ_s), are sensitive to temperature variations. This indicates that temperature plays a significant role in influencing the molecular dynamics and polarization mechanisms within the materials.

- The estimation of thermodynamic parameters using dielectric measurements provides valuable insights into the energy dynamics and molecular interactions within the blends and composites. Understanding these parameters is essential for optimizing material compositions and processing
- T_g values are determined using both dielectric measurements (DM) and differential thermal analysis (DTA) techniques. These methods are commonly used to analyze the thermal properties of materials, including their glass transition temperatures
- Char yield at 800°C and T_f improve with the inclusion of reinforcement materials
- Conditions, particularly in applications where thermal stability and energy management are critical factors.

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CONFLICTS OF INTEREST:

The author is asserting that their research was conducted impartially and without any external influences that could bias the results or conclusions.

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