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Research Article

Study on Thermal, Mechanical and Dynamic Properties of Epoxy Matrix with Hybrid of Nanoclay/Carbon Nanotube

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ABSTRACT

Hybrid polymer nanocomposites (HPNCs) represent a promising class of materials for various engineering applications. This study investigates the impact of combining nano clay (NC) and carbon nanotubes (CNTs) on the thermal and mechanical properties of epoxy matrices. Mechanical and thermal properties of HPNCs were characterized using TGA, DMTA, TMA, and SEM, and tested for flexural, tensile, shear, hot plate, and modal analyses. The obtained results were compared with similar theoretical ones from the Halpin-Tsai method. Results indicate that incorporating up to 5 wt % of NC alongside epoxy/CNT nanocomposites enhances CNT dispersion, leading to improvements in degradation temperature (+1%), glass transition temperature (+10%), thermal stability (50% increase in residual ash), storage modulus in the plastic range (+39%), transverse bond density (+41%), thermal expansion coefficient (-17%), flexural strength (+17%), elastic modulus (+85%), shear modulus (+11%), and natural frequency of the beam (+42%).

1. Introduction

Carbon nanotubes (CNTs) have garnered significant attention as additives in polymer composites due to their exceptional mechanical and electrical properties. Even a small amount of CNTs can drastically improve the performance of a polymer matrix. However, the agglomeration of CNTs, caused by strong van der Waals forces between individual nanotubes, remains a major drawback. This tendency to form bundles restricts their uniform dispersion, thereby limiting their effectiveness in polymeric nanocomposites. Numerous physical and chemical strategies have been developed to improve CNT dispersion, but these often compromise CNTs' inherent structure, leading to weakened properties in the nanocomposite. Studies have shown that adding small amounts of NC (typically less than 10%) to polymer matrices enhances various properties, including modulus, gas permeability, flame retardancy, and thermal stability [1-3]. This synergistic effect between CNT and NC is advantageous because NC, unlike surfactants or polymeric dispersants, does not harm the performance of CNTs. For instance, Liu et al. [2] reported that NC significantly improved the dispersion of CNTs in epoxy matrices,

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transforming CNT aggregates into continuous three-dimensional networks, which enhanced both the electrical and mechanical properties of the composite. Ayatollahi et al. [4] investigated the simultaneous use of NC and CNTs in epoxy nanocomposites and observed that adding just 5 wt.% NC to CNT/epoxy composites increased the elastic modulus and fracture toughness, although there was a slight reduction in ultimate strength. Similarly, Wang et al. [5] showed that growing CNTs on modified silicate plates led to significant enhancements in mechanical strength, hardness, and impact properties. In hybrid composites, the introduction of both CNT and NC has been shown to improve mechanical, thermal, and flame-retardant properties. Salimi et al. [6] demonstrated significant improvements in the mechanical properties of glass/carbon fiber-reinforced epoxy composites when CNTs and NC were used as hybrid fillers. Furthermore, Hosur et al. [7] found that the combined addition of CNTs and NC increased the crosslink density of epoxy nanocomposites, which translated into better thermal and mechanical performance. They also reported a 30% reduction in the coefficient of thermal expansion before the glass transition temperature. Recent studies have further confirmed these synergistic effects. Nguyen et al. [8,9] highlighted the significant improvements in tensile strength, modulus, fire resistance, durability ,and thermal stability in CNT/NC hybrid nanocomposites, attributing these enhancements to the efficient load transfer and uniform dispersion of the nanofillers. Additionally, Khosla et al. [10] demonstrated that hybrid nanocomposites incorporating CNTs and NC exhibited improved fatigue resistance and toughness for high-performance applications. The hybrid reinforcement not only mitigates the agglomeration of CNTs but also provides a barrier against thermal degradation, improving the overall stability of the composite. A study by Maurya et al. [11] explored the mechanical properties of hybrid nanocomposites and found a direct correlation between CNT agglomeration and mechanical performance. This indicates that further investigation into the optimization of filler concentrations could yield significant benefits in the design of advanced composite materials. Furthermore, Rezaei Qazviniha and Piri [12] examined the impact of nanoparticle concentration on mechanical properties, reinforcing the importance of understanding filler interactions in enhancing material performance. An innovative theorical research approach to enhance CNT dispersion without compromising their integrity involves the incorporation of nanoclay (NC). NC not only aids in CNT dispersion but also serves as a reinforcing agent that improves the mechanical and thermal properties of the polymer matrix [13,14]. Due to its stiff, plate-like structure, NC can prevent the re-aggregation of CNTs, creating a more uniform nanocomposite structure. Additionally, NC is a cost-effective material that offers unique electrochemical properties, enhancing the performance of CNT composites. Its negatively charged surface also helps establish better interactions with polymer matrices, particularly in CNT nanocomposites [15]. Barkoula, et.al [16] discuss the beneficial effects of adding nano clay on the dispersion of carbon nanotubes (CNTs) in their study on flame-retardant polymer composites. They concluded that the incorporation of nanoclay significantly enhances CNT dispersion, addressing issues of agglomeration and resulting in improved mechanical and thermal properties of the composites. Tuan Anh Nguyen et al.[17] concludes that incorporating nano clay and multi-walled carbon nanotubes into epoxy resin significantly enhances both the mechanical properties and flame retardancy of the resulting nanocomposites. This combination leads to improved tensile strength and thermal stability, making these materials suitable for applications requiring fire resistance and structural integrity. This study investigates the effects of varying amounts of NC on the mechanical and thermal properties of CNT/epoxy nanocomposites. The synergistic effects of these two nanofillers are explored through a comprehensive analysis of thermal, mechanical, and morphological characteristics to develop a high-performance hybrid polymer nanocomposite. Finally, the comparison is made with the Three-phase Halpin Tsai model [13,14].

The present study aims to systematically investigate the effects of incorporating varying amounts of NC alongside carbon nanotubes (CNTs) within an LY 5052 epoxy matrix. This research is motivated by the need to enhance the thermal and mechanical properties of hybrid polymer nanocomposites (HPNCs) for advanced engineering applications. By employing a range of characterization techniques—including thermogravimetric analysis (TGA), dynamic mechanical thermal analysis (DMTA), thermomechanical analysis (TMA), scanning electron microscopy (SEM), and comprehensive mechanical testing—the study seeks to establish a clear relationship between the filler content and the resulting material properties. Specifically, it focuses on key parameters such as degradation temperature, glass transition temperature, elastic modulus, flexural strength, and flame retardancy. Ultimately, the findings are expected to contribute valuable insights into the optimization of HPNCs based on LY 5052 epoxy, paving the way for their use in high-performance applications. The below table provide a simplification of comparison between the current study and previous researches:

Study	Materials	Methods	Outcomes (Improvement in Properties)
Liu et al. [2]	CNTs, NC, epoxy	Enhanced CNT dispersion using NC	Tensile strength (+30–50%), electrical conductivity (+35%), and enhanced morphological dispersion of CNTs due to NC's ability to transform CNT aggregates into networks
Ayatollahi et al. [4]	CNTs, NC, epoxy	Simultaneous inclusion of NC and CNTs	Elastic modulus (+15–20%), fracture toughness (+10–15%), and ultimate strength (-5–10%) due to NC aiding CNT dispersion and reinforcing the matrix
Wang et al. [5]	CNTs, modified silicate plates	Growing CNTs on silicate plates	Mechanical strength (+30%), hardness (+25%), and impact resistance (+35%) due to aligned CNT growth on silicate plates enhancing filler-matrix bonding
Salemi et al. [6]	CNTs, NC, glass/carbon fiber epoxy	Hybrid filler in fiber-reinforced composites	Flexural strength (+20–30%), tensile modulus (+25%), impact resistance (+30%) due to synergistic effects of CNT and NC
Hosur et al. [7]	CNTs, NC, epoxy	Combined CNT and NC inclusion	Thermal stability (+20–25%), elastic modulus (+25%), fracture toughness (+20%), and coefficient of thermal expansion (-30%)
Nguyen et al. [18]	CNTs, NC	Hybrid nanocomposite fabrication	Tensile strength (+25–35%), tensile modulus (+30%), and thermal stability (+20%) due to improved load transfer and uniform dispersion
Khosla et al. [10]	CNTs, NC	Hybrid nanocomposite development	Fatigue resistance (+20–30%), fracture toughness (+20–30%), and thermal stability (+15–20%) enhanced by hybrid filler interactions
Pinto et al. [31]	CNTs, NC	Investigated CNT agglomeration correlation	Suggested optimization of filler concentrations could lead to tensile strength (+15–25%) and elastic modulus (+20%)
Rezaei Qazviniha & Piri [12]	CNTs, NC	Examined nanoparticle concentration effects	Emphasized filler interaction for fracture toughness (+20–30%), elastic modulus (+25%), and thermal stability (+20%)
Present Study	CNTs, NC, LY5052 epoxy	Various characterization techniques (TGA, DMTA, TMA, SEM, and mechanical tests)	Degradation temperature (+1%), glass transition temperature (+10%), thermal stability (+50% residual ash), storage modulus (+39%), transverse bond density (+41%), thermal expansion coefficient (-17%), flexural strength (+17%), elastic modulus (+85%), shear modulus (+11%), and natural frequency of beam (+42%)

2. Experimental

2.1. Materials

This study utilizes three primary materials: carboxyl functionalized multi-walled carbon nanotubes (MWCNTs), LY 5052 epoxy resin with HY 5052 hardener, and Montmorillonite K10 nanoclay, each contributing distinct advantages to the hybrid nanocomposites.

LY 5052 Epoxy Resin: Manufactured by Huntsman, this epoxy resin is combined with HY 5052 hardener at a mass ratio of 100:38. The 100:38 mass ratio of epoxy to hardener ensures optimal curing by allowing complete crosslinking between reactive groups, which maximizes mechanical and thermal properties. Following this ratio is essential for achieving the intended strength, stability, and durability of the epoxy matrix. Epoxy resins are favored for their excellent adhesion, high strength, low shrinkage, and thermal stability, making them compatible with CNTs and nanoclays. Research indicates that the interaction between epoxy matrices and nanofillers leads to enhanced mechanical and thermal properties [7, 8].

Carboxyl Functionalized MWCNTs: These MWCNTs, with an average diameter of 20–30 nm and a length of 10–30 µm, were procured from U.S. Research Nanomaterials, Inc. MWCNTs are recognized for their exceptional mechanical, electrical, and thermal properties. Their high aspect ratio and surface area contribute to improved strength, conductivity, and stability in polymer matrices. However, their tendency to agglomerate can hinder performance, which is mitigated by functionalization with carboxyl groups to enhance dispersion and interaction with the epoxy [1, 3, 6].

Montmorillonite K10 Nanoclay: With a purity of 99.9% and a surface area of 220–270 m^2/g, this nanoclay serves as a secondary reinforcing agent. Its plate-like structure prevents CNT re-agglomeration and enhances the mechanical and thermal properties of composites. The negatively charged surface of nano clay promotes strong interactions with epoxy matrices, further improving composite performance [2, 4, 5].

It is crucial to understand the rationale behind the selection of nanofiller percentages in this work, as this determines the balance between improved performance and potential issues such as agglomeration or saturation. Properly hoosing filler concentrations ensures optimized enhancement of the thermal, mechanical, and structural properties of the composite material without compromising its integrity. Incorporating 0.5% of carbon nanotubes (CNTs) into nanocomposites has been widely recognized as an optimal concentration for enhancing mechanical and thermal properties. This balance allows for improved dispersion, resulting in fewer agglomeration issues, which are common at higher concentrations. Studies have shown that 0.5% CNTs significantly improve mechanical properties like tensile strength and modulus while maintaining effective distribution within the polymer matrix. For example, Ayatollahi et al. [4] demonstrated significant improvements in mechanical and electrical properties at this concentration, while Salimi et al. [6] reported enhanced flexural and tensile strength in hybrid nanocomposites without the drawbacks of excessive CNT clustering. Similarly, Maurya et al. [11] concluded that 0.5% CNT provided the best mechanical performance with minimal particle agglomeration. These findings suggest that 0.5% CNT is an ideal concentration for enhancing nanocomposite properties without compromising material integrity due to clustering. The rationale for selecting nanoclay (NC) concentrations of 1%, 3%, 5%, and 7% is based on studies that have shown varying effects on the properties of nanocomposites. Lower concentrations (1% and 3%) enhance dispersion and moderate improvements, while 5% can optimize mechanical and thermal characteristics. However, at 7%, particle agglomeration occurs, which can weaken the composite. Several works in the literature corroborate these findings, emphasizing the optimal dispersion and mechanical enhancements at 5% NC loading. For instance, Salimi et al. [6] showed improvements in mechanical properties up to 5% NC in hybrid composites with CNT and nano clay fillers. Maurya et al. [11] found that 5% NC provided optimal thermal and mechanical performance, while higher concentrations led to agglomeration. Rezaei Qazviniha and Piri [12] observed particle agglomeration at NC levels above 5%, weakening the composite structure. Khosla et al. [10] found that incorporating MWCNTs and nanoclay fillers enhanced both tensile modulus and thermal stability, with 5 wt% nanoclay providing the best balance between mechanical and thermal improvements. The novelty of this study lies in its investigation of the synergistic effects of carbon nanotubes (CNT) and nano clay in epoxy nanocomposites, specifically addressing their combined influence on mechanical properties and fire resistance. While previous research has focused on individual contributions of these materials, this work provides new insights into optimizing formulations for enhanced performance, thereby advancing the understanding of composite materials for a range of applications.

2.2 Preparation of Hybrid of Epoxy/ CNT/NC

Before preparing the nanocomposite, silicon molds are made according to the dimensions of the standards required to perform laboratory tests. The polymeric hybrid nanocomposite samples were composed via a mixing and casting method, as shown in Fig.2 which consists of the below stages [7].

Step 1. Weight the fillers, epoxy, and hardener, and Mix the defined mass fractions of nanofillers into the hardener.

Step 2. Put the resulting combination in an ice bath and blend by using an ultra-sonication (60% Ampl) for 30 min.

Step 3. Pour the epoxy into the resulting blend and use a mechanical shaft in an ice bath (900 rpm) for 5 min to gain a smooth combination.

Step 4. Degas by an ultrasonic ice bath for one hour.

Step 5. Cast the final mixture in pre-pared silicone molds. Place at room temperature for 24 hours to cure.

Step 6. Post-cure in the oven at 100˚C for 4 hours.

Now the samples are ready to accomplish the demanded experiments.

2.3 Three-phase Halpin-Tsai method

The three-phase Halpin-Tsai is a micromechanical model used to predict the mechanical properties of multi-phase composites. It operates in two stages(see **Error! Reference source not found.**): initially, the neat matrix (such as epoxy) is mixed with the first filler (e.g., carbon nanotubes) to form a reinforced matrix. In the second stage, a second filler (e.g., nano clay) is added to this new matrix, resulting in the final composite material. Previous studies have validated this approach for multi-phase composites ([9, 18]). Halpin-Tsai method expression is as follows:

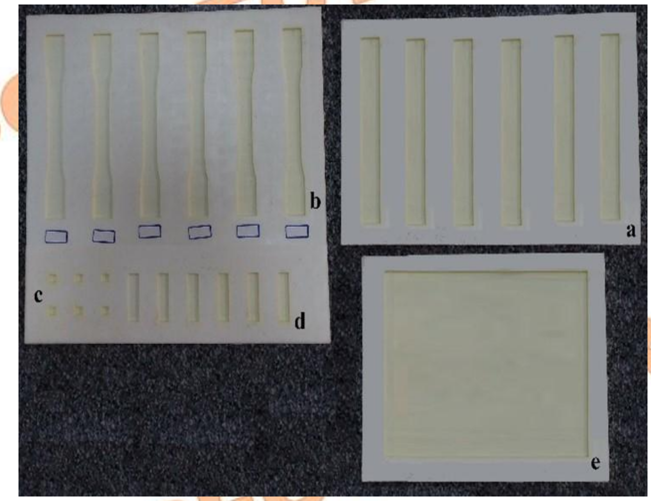

Fig.1 Silicon mold

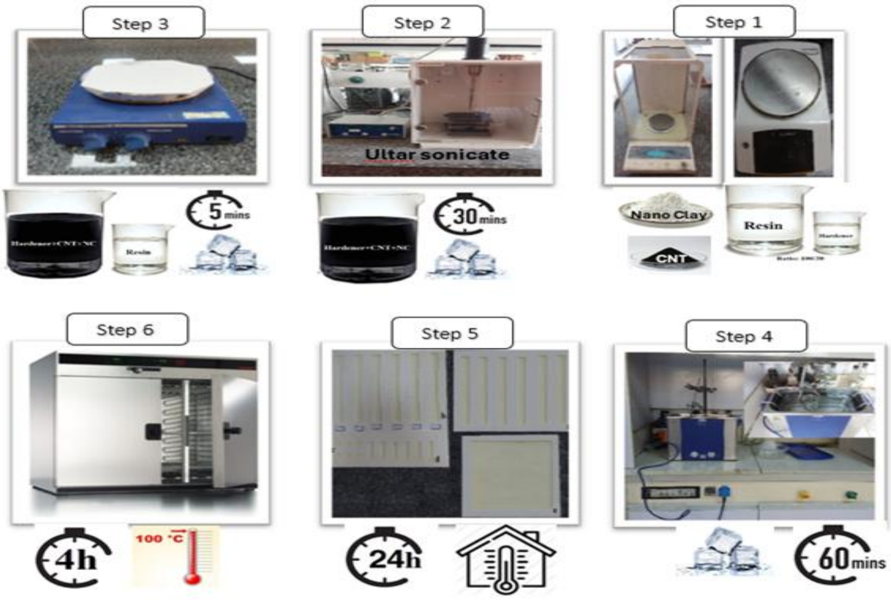

Fig.1: Construction Steps [7].

$$\frac{M}{M_m}=\frac{1+\alpha\eta\phi_f}{1-\eta\phi_f} \quad , \quad \eta=\frac{M_f/M_m-1}{M_f/M_m+\alpha} \tag{1}$$

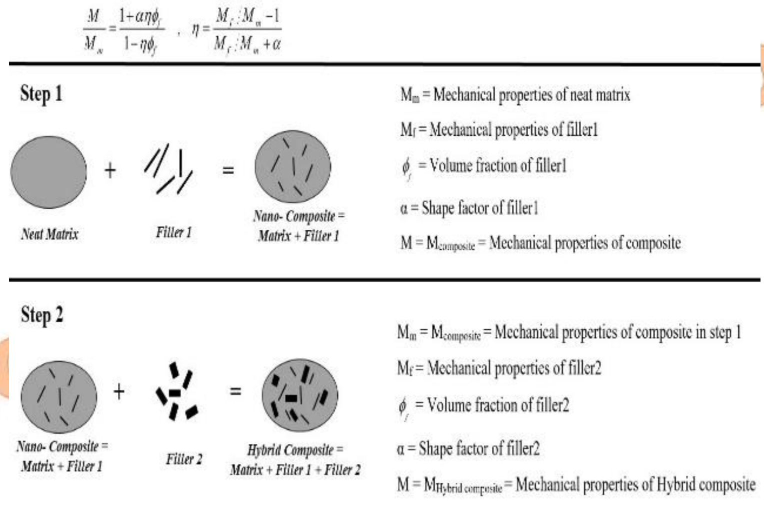

Fig.3. A schematic of how to apply the three-phase Halpin Tsai method[13,14]

In Eq.(1), ϕ_f is the volume fraction of nanofiller, M is the mechanical feature, indices f and m present filler and matrix, respectively. Three-phase Halpin-Tsai includes two stages: first, the neat matrix and the filler 1 are mixed and used as a new matrix, then the filler 2 is added to the new matrix (**Error! Reference source not found.**.3).

3. Results and Discussion

This section will present the experimental outcomes derived from the thermal and mechanical characterization of hybrid nanocomposites, consisting of epoxy, carbon nanotubes (CNTs), and nano clay (NC) at different weight percentages (1%, 3%, 5%, and 7%). The findings will focus on key parameters, such as thermal stability, mechanical strength, and microstructural features, all examined using TGA, DMTA, FE-SEM, and mechanical testing techniques. The influence of NC concentration on the dispersion and agglomeration of CNTs, as well as its effects on the enhancement of the composites' overall performance, will be discussed. Additionally, comparisons with the Halpin-Tsai theoretical model will be made to evaluate the agreement between experimental results and predictive models. The section aims to highlight the optimal NC concentration for improved nanocomposite performance while identifying possible trade-offs or limitations observed during testing.

3.1. Field-Emission Scanning

To examine the fracture surfaces of the samples under study, FE-SEM analysis was performed, and the results are presented in Fig. 2. The red circles highlight the areas of agglomeration, while the red arrow points to the carbon nanotubes (CNTs). In Fig. 2 a-d, it is evident that CNTs tend to cluster into micro-sized particles in certain regions of the polymeric matrix. However, as the weight percentage (wt.%) of nanoclay (NC) increases, the occurrence of these agglomeration regions progressively decreases.

The FE-SEM micrographs provide a detailed view of the fracture surfaces of epoxy-0.5% CNT nanocomposites with varying nano clay (NC) concentrations (0% to 7%).

(a) 0% NC: The micrograph in Fig. 2a shows the epoxy matrix with 0.5% CNT but no nano clay. Numerous CNT agglomerated regions are visible, indicating poor dispersion. The relatively smooth fracture surface suggests brittle fracture behavior typical of neat epoxy composites with insufficient filler dispersion.

(b) 1% NC: Comparing Fig. 2a and b, the introduction of 1% NC reduces the number of CNT agglomerated regions and increases the presence of separated CNTs. The fracture surface

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becomes rougher, indicating improved energy dissipation and a potential increase in toughness. However, some CNT-NC clusters are still present, pointing to the initial stages of improved filler dispersion.

(c) 3% NC: By increasing the NC concentration to 3wt.% (Fig. 2c), there is a significant reduction in both the number and size of agglomerated CNT regions. The interaction between CNTs and NC improves, and the fracture surface shows increased roughness, suggesting better matrix-filler bonding and enhanced mechanical properties.

(d) 5% NC: At 5% NC (Fig. 2d), the agglomeration area of CNTs decreases further, and more dispersed CNTs are visible. The rough and irregular fracture surface implies optimal energy dissipation and suggests that 5% NC promotes the best nanoparticle dispersion. This optimal concentration likely contributes to the most substantial improvement in the physical and mechanical properties of the composite.

(e) 7% NC: However, with a further increase in NC to 7wt.% (Fig. 2e), new weak regions start to form due to the excessive agglomeration of NC in the matrix. While the surface still shows irregularities, indicating fracture toughness, the large clusters of NC hinder uniform stress distribution, potentially compromising the composite's mechanical performance.

The FE-SEM analysis from this study shows that introducing nano clay (NC) into the epoxy-0.5% CNT nanocomposite significantly improves CNT dispersion and reduces agglomeration. This improvement is most evident at 5% NC, where the optimal dispersion of CNTs results in enhanced mechanical and thermal properties. However, at concentrations higher than 5% NC, such as 7%, nanoparticle agglomeration occurs, leading to weak regions in the matrix and negatively affecting the overall performance.

3.2. Thermal Gravimetric Analysis(TGA)

Thermogravimetric analysis (TGA) is a powerful technique used to evaluate the thermal stability and decomposition characteristics of materials by measuring weight changes as a function of temperature or time. The results are shown in **Error! Reference source not found.** and **Error! Reference source not found.** The addition of 0.5 wt.% CNT to the epoxy (EP) matrix lowered the onset and degradation temperatures due to CNT's high thermal conductivity, which accelerates heat distribution and leads to earlier degradation. This aligns with Wang et al. [5], who found that CNTs reduce thermal stability by transferring heat faster. However, adding nanoclay (NC) up to 5 wt.% reversed this effect, increasing both temperatures by creating a physical barrier that delays curing and strengthens the EP-NC network, requiring more energy to degrade. Hosur et.al.[7] similarly observed NC delaying epoxy hybrid curing. When NC concentration reached 7 wt.%, thermal stability decreased due to nanofiller agglomeration [7].

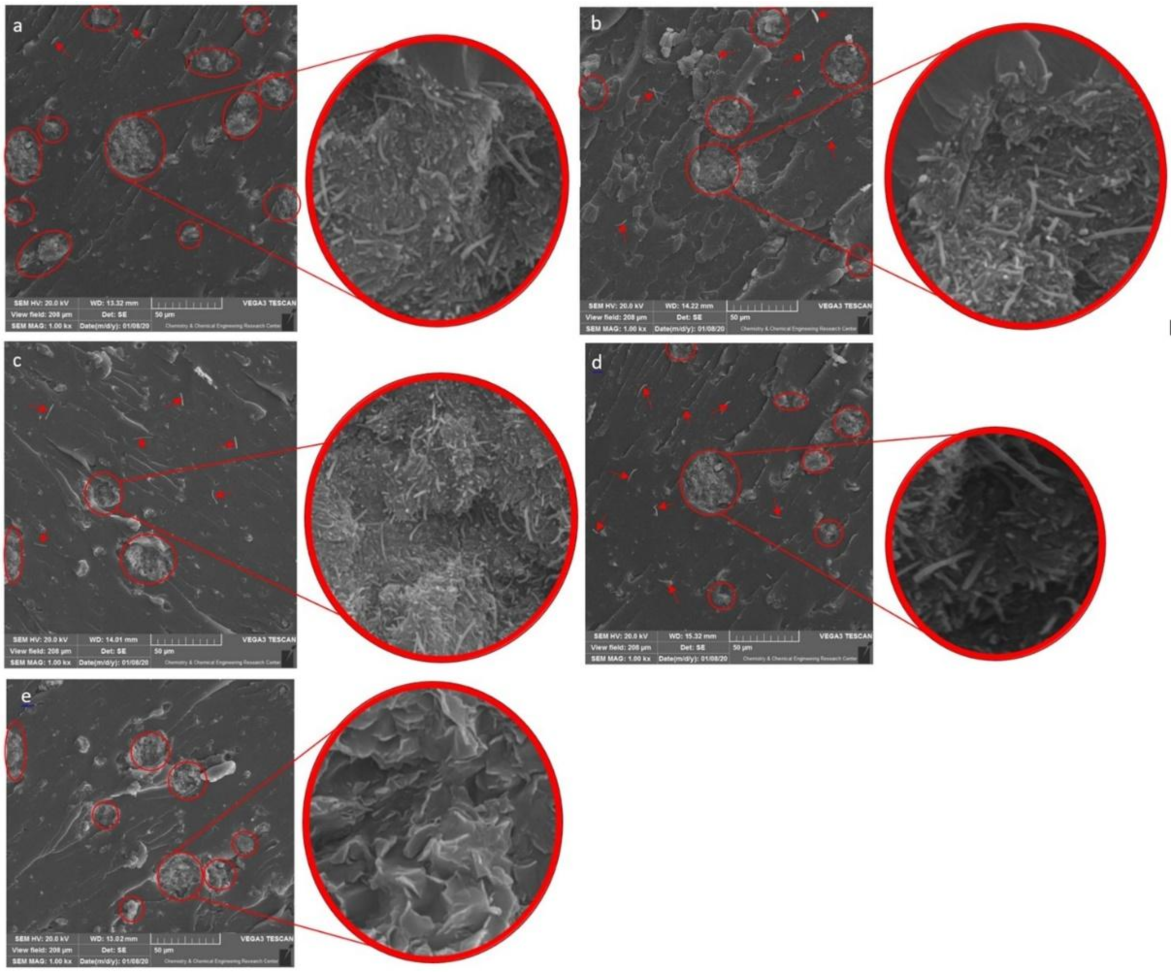

Fig. 2: Field-Emission Scanning Electron Microscope (FE-SEM) analysis (with magnification 50 µm) of epoxy-0.5% CNT containing (a) 0%NC, (b) 1%NC, (c) 3%NC, (d) 5%NC, and (e) 7% NC

Table 1: The obtained results from the TGA/DTG analysis

Sample	Onset temperature (°C)	Degradation temperature (°C)	Remained ash (%)
EP	258.5	325.8	12.48
EP-0.5%CNT	257.5	325.0	13.44
EP-0.5CNT-1%NC	262.5	326.7	14.61
EP-0.5%CNT-3%NC	265.5	327.5	15.44
EP-0.5%CNT-5%NC	273.2	329.1	18.73
EP-0.5%CNT-7%NC	270.6	330.2	18.42

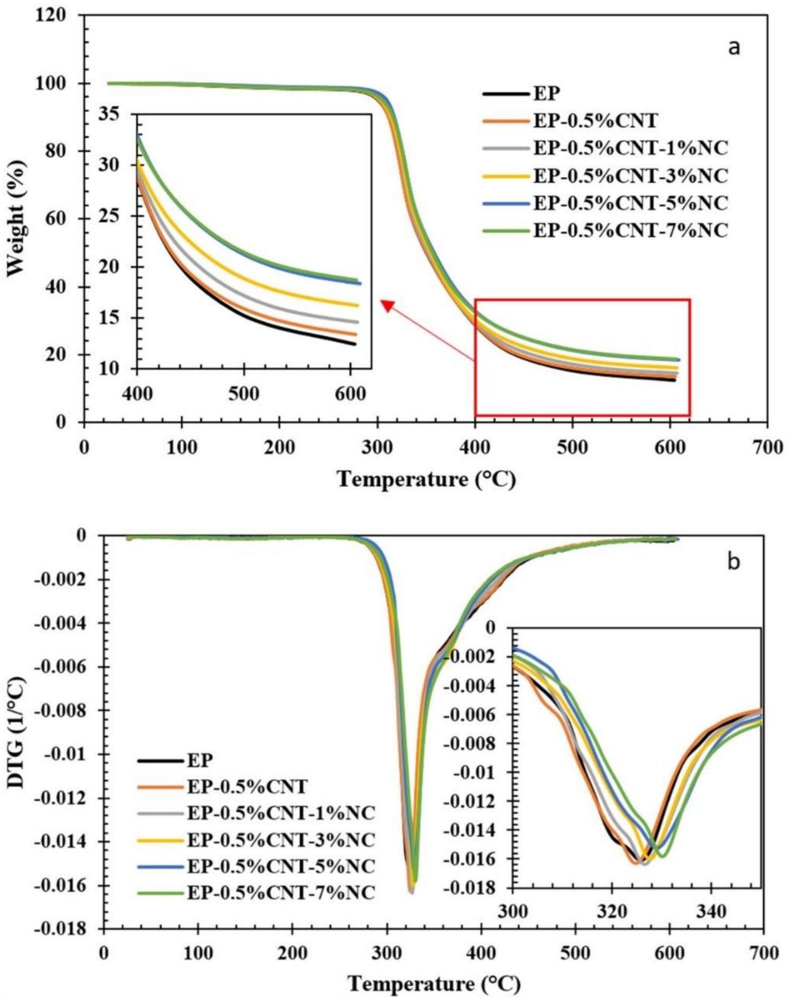

Fig. 3: TGA/DTG thermograms of pure EP and EP containing 0.5wt.% CNT in the absence and presence of different concentrations of NC nanoparticles

The remained ash percentage increased with the addition of CNTs due to their superior thermal stability compared to the epoxy matrix, consistent with Geng et al. [15], who found that CNTs raise residual ash content. Ash content continued to rise with NC concentrations up to 5 wt.%, likely because NC improved CNT dispersion and enhanced thermal resistance, as noted by Maurya et al. [11]. However, at 7 wt.% NC, the remained ash percentage decreased, attributed to nanofiller agglomeration that weakened the matrix. This observation aligns with Hosur et al. [7], who also reported reduced thermal stability and ash content due to the clustering of nanofillers at higher concentrations.

The overall thermal stability of the nanocomposites improved as NC concentration increased up to 5 wt.%, likely due to better dispersion of CNTs and the formation of a strong EP-NC network. This network, combined with the high thermal stability of CNTs, increased the composite's resistance to degradation. However, as NC content reached 7 wt.%, the thermal stability decreased due to filler agglomeration, which diminished the effectiveness of the composite. This trend is supported by the work of Salimi et al. [6], who found that while lower concentrations of NC and CNTs enhance thermal properties, higher concentrations lead to filler agglomeration, reducing overall performance. These results underscore the importance of optimizing filler concentrations to balance dispersion and thermal performance in hybrid nanocomposites.

3.3. Dynamic Mechanical Thermal Analysis(DMTA)

DMTA was conducted to evaluate the thermal and mechanical properties of the epoxy matrix

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reinforced with carbon nanotubes (CNTs) and nano clay (NC) in hybrid nanocomposites. This technique measures the variation in storage modulus (E’) and loss modulus (tan δ) as a function of temperature, revealing the material's stiffness, glass transition temperature (Tg), and cross-linking density (υe). Table 2 and **Error! Reference source not found.** show the result of DMTA.

●*Storage Modulus (E’) Results*

The storage modulus (E’) of pure epoxy (EP) is the lowest across the temperature range, indicating its inherent lower stiffness. The addition of 0.5 wt.% CNT to the epoxy matrix (EP-0.5% CNT) leads to an increase in the storage modulus, suggesting that CNTs reinforce the matrix due to their high aspect ratio and excellent mechanical properties. CNTs improve load transfer within the matrix, as highlighted by Ayatollahi et al. [4].

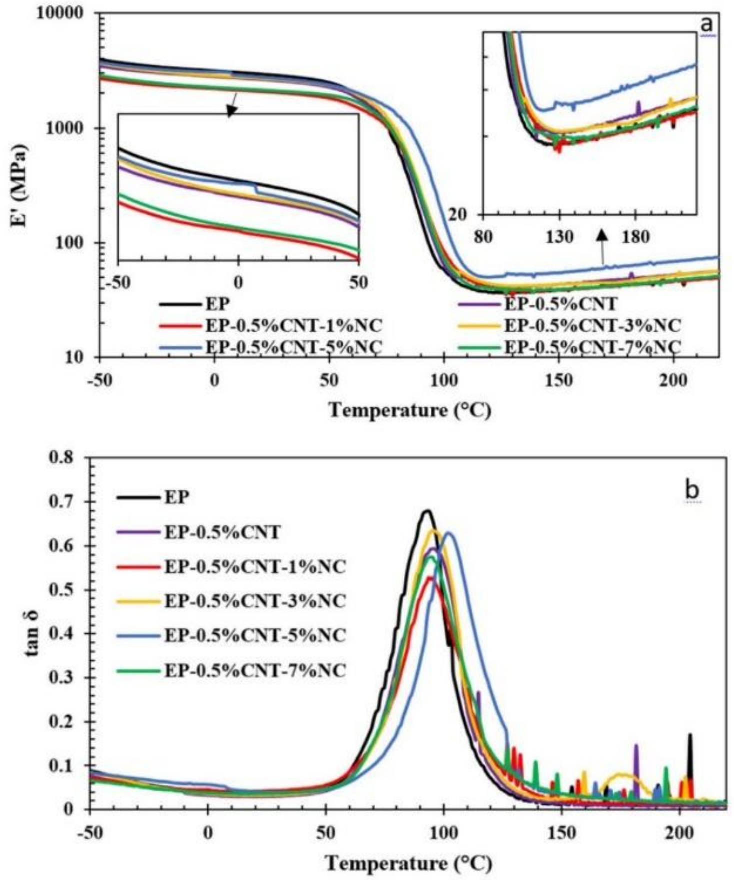

Fig.6. Variations of (a) storage modulus and (b) loss peak (tan δ) of the studied samples against the applied temperature

Table 2: DMTA test results for the pure EP, and EP containing 0.5 wt.% CNT in the absence and presence of different NC concentrations

Sample	T_g (°C)	Storage modulus (MPa)		υ_e (mol/m^3)
		At glassy region	At rubbery region	
EP	93.04	3944.8	36.5	0.0037
EP-0.5%CNT	96.90	3361.8	40.2	0.0041
EP-0.5CNT-1%NC	96. 30	2591.4	37.7	0.0035
EP-0.5%CNT-3%NC	96.60	3511.2	41.8	0.0042
EP-0.5%CNT-5%NC	102.50	3670.2	50.6	0.0052
EP-0.5%CNT-7%NC	95.20	2755.8	39.1	0.0041

- *Glass Transition Temperature (Tg) and Loss Modulus (tan δ)*

The glass transition temperature (Tg), indicated by the peak of the tan δ curve, represents the

transition from a rigid to a more flexible material. Pure epoxy exhibits the lowest Tg around 100°C, indicating higher chain mobility and lower cross-linking density. The incorporation of 0.5 wt.% CNT into the matrix (EP-0.5% CNT) increases the Tg due to restricted molecular mobility around the CNTs, which reduces the free volume in the matrix. Ayatollahi et al. [4] observed similar effects, confirming the increase in Tg with CNT addition. The inclusion of NC further raises the Tg as its concentration increases up to 5 wt.% (EP-0.5% CNT-5% NC). This increase is due to the physical barrier effect of NC, limiting the mobility of epoxy monomers and enhancing the cross-linking network. Rezaei Qazviniha and Piri [12] showed that NC nanoparticles significantly affect the Tg of hybrid nanocomposites, improving their thermal properties. At 7 wt.% NC, the Tg decreases slightly (EP-0.5% CNT-7% NC). This reduction can be attributed to nanoparticle agglomeration, which decreases effective interactions between the matrix and the fillers. Salimi et al. [6] mentioned that high filler loadings can lead to clustering, reducing the reinforcing efficiency of the fillers.

- *Cross-Linking Density (υe)*

Cross-linking density (υe) reflects the rigidity of the nanocomposite network structure. It was calculated based on the storage modulus in the rubbery region as follows): [12].

$$\upsilon_e = E'/3RT \qquad (2)$$

where E' is the minimum storage modulus at the rubbery region. R and T are universal gas constant and absolute temperature, respectively.

Pure epoxy exhibits the lowest cross-linking density, corresponding to its lower storage modulus and Tg. The addition of 0.5 wt.% CNT increases the cross-linking density due to CNTs acting as cross-linking points in the matrix. This is consistent with observations by Ayatollahi et al. [4], who noted an increase in cross-linking density in CNT-reinforced nanocomposites due to enhanced matrix-filler interaction. Including NC in the hybrid nanocomposite further raises the cross-linking density, peaking at 5 wt.% NC (EP-0.5% CNT-5% NC). The improved dispersion of CNTs in the presence of NC results in a denser cross-linking network, as confirmed by Maurya et al. [11]. The enhanced network structure improves the thermal and mechanical properties of the composite. However, at 7 wt.% NC, the cross-linking density decreases slightly, likely due to nanoparticle agglomeration. This behavior is in line with the decreased Tg and storage modulus at higher NC content, demonstrating that excess NC leads to poor dispersion and ineffective cross-linking, as mentioned by Salimi et al. [6].

3.4. Thermomechanical Analysis(TMA)

To evaluate thermal expansion coefficient values, thermomechanical analysis (TMA) was accomplished, and the related curves are depicted in Fig.4 . The parameters obtained from the TMA curves are summarized in Table 3. In Table 3, α_1 and α_2 represent the thermal expansion coefficients in the lower and higher temperature ranges than the glass transition temperature (Tg), respectively. The Tg values before and after the addition of nanoclay (NC) at different concentrations varied between 78°C and 89°C. Although the Tg values obtained from TMA differ from those acquired through dynamic mechanical thermal analysis (DMTA), a similar trend of increase is observed, indicating that the addition of NC nanoparticles enhances the stiffness of the nanocomposite [19]. This observation aligns with findings from previous studies; for instance, Khosla et al. [10] reported that the introduction of NC to polymer matrices improves thermal stability and reduces thermal expansion due to the restricted mobility of polymer chains. Additionally, the values for α_1 generally decreased (except for the EP-0.5CNT-1NC sample), while α_2 exhibited an increase (except for the EP-0.5CNT-5NC sample) following the incorporation of clay nanoparticles into the nanocomposite. Consequently, no clear trend can be established regarding the variation of α_1 and α_2 values with different nanoparticle concentrations. However, as indicated in Table 3, the EP-0.5CNT-5NC sample exhibited the lowest thermal expansion coefficient among the samples, while the EP-0.5CNT-1NC sample had the highest thermal expansion coefficient. The reported Tg values in this study (78°C to 89°C) are consistent with those found in earlier research. Salimi et al. [6] observed that incorporating small amounts of NC leads to an increase in Tg, supporting the findings of enhanced stiffness and structural integrity in the composites. This further corroborates the notion

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that the presence of NC effectively contributes to the thermal performance of the composite, validating the current results. Moreover, the observed behavior of thermal expansion coefficients, with a decrease in α_1 and an increase in α_2 (except for certain samples), reflects findings from Maurya et al. [11], who noted similar changes in hybrid nanocomposites. They attributed these variations to the complex interactions between the polymer matrix and the nanofillers, affecting free volume and, consequently, thermal expansion behavior.

In summary, the thermomechanical properties of the hybrid polymer nanocomposites, as demonstrated through TMA, confirm the significant influence of NC on enhancing thermal stability and modifying thermal expansion characteristics. The findings presented here are consistent with previous literature, supporting the validity of the observed trends and establishing a strong foundation for continued exploration of hybrid nanocomposites in advanced engineering applications.

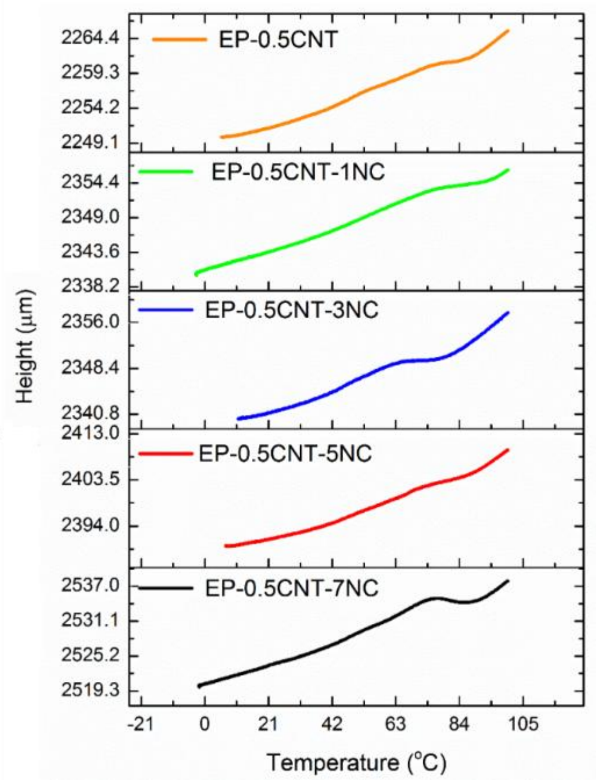

Fig.4: TMA curves of EP containing 0.5wt.% CNT in the absence and presence of different concentrations of NC nanoparticles

Table 3: The thermal expansion parameters from the TMA curves

Sample	T_g (°C)	α^1(/°C)	α^2(/°C)
EP	88.5	102.50	81.17
EP-0.5CNT-1%NC	78.01	98.0	155.6
EP-0.5%CNT-3%NC	71.88	79.5	144.3
EP-0.5%CNT-5%NC	89.02	73.73	84.5
EP-0.5%CNT-7%NC	78.02	75.69	123.8

3.5 Felextural Test

The three-point bending flexural tests were conducted in accordance with ISO 178-2019, using specimens with dimensions of 125 × 12.7 × 3.2 mm at a crosshead speed of 2 mm/min.

- *Flexural Strength (Fig. 5 (a))*

Fig. 5a illustrates that the flexural strength of the epoxy matrix increases with the addition of a small amount of carbon nanotubes (CNTs). This enhancement in strength is primarily due to improved interfacial adhesion and dispersion of CNTs within the epoxy matrix, both critical factors in strengthening the composite material [1, 2, 4]. Several studies confirm that well-dispersed CNTs facilitate better load transfer within the matrix, which contributes to the observed improvement in flexural strength [3, 5]. The introduction of nanoclay (NC) to the epoxy/CNT nanocomposite further enhances CNT dispersion, reducing agglomeration and promoting a more uniform distribution. This effect helps to improve the mechanical properties by stabilizing CNTs within the matrix [6, 7]. However, as observed in this Figure, increasing NC content beyond an optimal point begins to reduce the flexural strength.

This decline is attributed to the aggregation of excess NC and CNTs, which leads to stress concentration points and weakens the composite structure, a phenomenon reported by Nguyen et al. [19] and Rezaei Qazviniha et al. [12].

- *Flexural Strain (Fig.* 5 *(b))*

Fig. 5b shows that the initial addition of 0.5 wt.% CNT decreases the flexural strain, indicating that CNTs add stiffness to the material, thus reducing its flexibility. This behavior is consistent with findings by Geng et al. [13], who reported that CNTs increase stiffness but may limit the polymer matrix's ability to deform under stress. Interestingly, when 1 wt.% NC is incorporated into the epoxy/CNT composite, the flexural strain increases slightly, suggesting that NC at lower concentrations helps alleviate brittleness by enhancing CNT dispersion. However, as more NC is added, the flexural strain begins to decrease, indicating a transition toward a more brittle structure. This result aligns with studies by Khosla et al. [10] and Gojny et al. [20], which show that excessive filler content can restrict matrix mobility, thereby reducing flexibility.

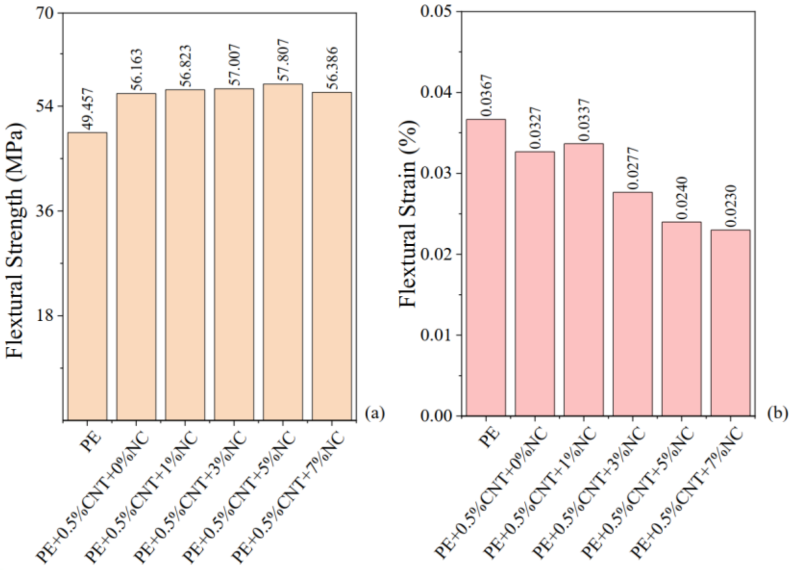

Fig. 5: (a) Flexural Strength and (b) Flexural strain

3.6 Tension Test

This section presents an analysis of the tensile properties of pure epoxy and various nanocomposites incorporating carbon nanotubes (CNT) and nano clay (NC), based on tensile tests conducted in accordance with ASTM D638 using type I specimens. The tests were performed on pure epoxy and six different nanocomposite formulations, each consisting of varying combinations of CNT and NC, with three samples analyzed for each composite type. The incorporation of nanoparticles into a polymer matrix can significantly alter its tensile properties through several mechanisms: The exceptional mechanical properties of nanoscale particles can enhance toughness and strength. Two-dimensional nanoparticles like NC can impede crack propagation, effectively hindering fracture. Structural variations in the macromolecular chains adjacent to the nanoparticle surfaces can influence the overall tensile properties.

The improvement of tensile properties in nanocomposites is closely linked to the degree of dispersion of the nanofiller within the matrix and the interfacial interactions between the nanofiller surfaces and the polymer [20,21]. As illustrated in Fig. 6a, which depicts the modulus of elasticity and percent strain to break for different nanofiller combinations, the results indicate that increasing NC content enhances the modulus of elasticity, which is attributed to improved CNT dispersion. Specifically, the addition of up to 5 wt.% NC to the CNT/epoxy nanocomposite increases the modulus of elasticity due to the following reasons:

NC layers are inherently stiff and possess a high modulus.

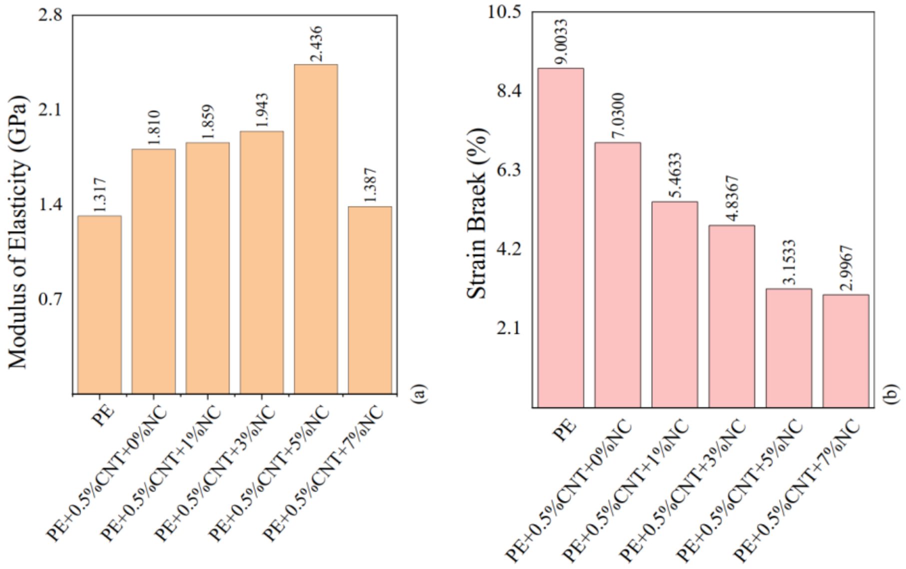

Fig. 6: (a) Modulus of elastic and (b) Strain break

The presence of intercalated and exfoliated NC within the matrix significantly increases the surface area exposed to the polymer, resulting in physical absorption and adherence that stiffens the nanocomposite. The concurrent use of NC alongside CNT in the epoxy matrix aids in improving the overall dispersion of CNT. In Fig. 6 b, it can be observed that the addition of 0.5 wt.% CNT results in a decrease in strain at break, while incorporating 1 wt.% NC into the CNT/epoxy nanocomposite leads to an increase in strain at the break by +1.6%. The observed decrease in strain at break may be attributed to the constrained mobility of polymer chains due to the presence of NC platelets. These results are consistent with earlier reports [8, 23], supporting the findings that the type and content of nanofillers play a significant role in enhancing the mechanical performance of epoxy nanocomposites. The Tensile testing was performed for each nanocomposite compound with three samples. The stress-strain diagram using the average data is shown in Fig.10.

●*Comparison with the three-phase Halpin-Tsai method*

In Fig.8, the comparison between the experimental elastic modulus and the theoretical predictions from the three-phase Halpin-Tsai method reveals important observations. The theoretical predictions closely align with the experimental results, particularly at 3% wt. of nanoclay (NC). This indicates the effectiveness of the Halpin-Tsai method in estimating the elastic modulus of the composite at this concentration level, as supported by previous research (e.g., [9, 18, 24]). The theoretical results in Fig.8 exhibit a linear trend, which can be attributed to the assumptions of the Halpin-Tsai model, including uniform distribution and effective load transfer between the matrix and fillers. The model considers the mechanical properties as a direct function of the volume fraction of fillers in a small amount of filler, leading to a linear relationship when there is no significant interaction or agglomeration between the fillers. This linear behavior is ideal for predicting the modulus in scenarios where filler particles are well-dispersed, as observed in earlier studies (references [18, 9]).

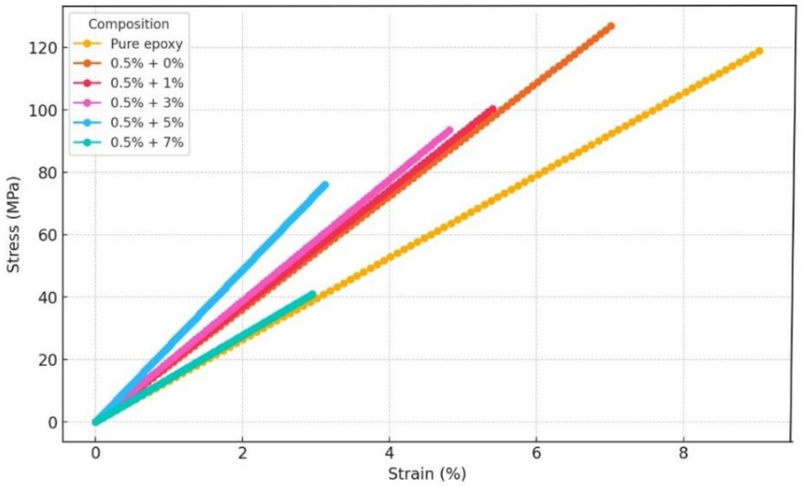

Fig. 7: Stress-Strain Diagram

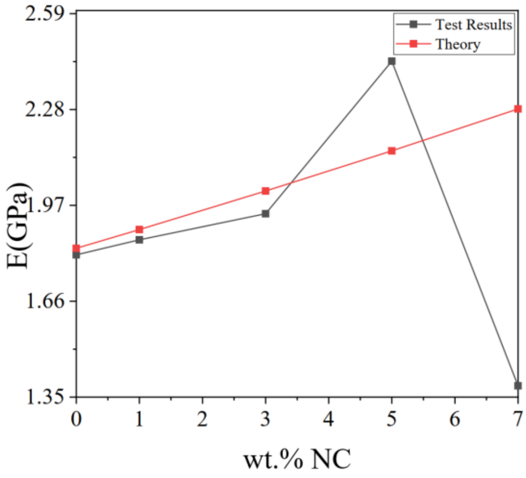

Fig.8: Comparison of experimental elastic modulus and theoretical results of the three-phase Halpin Tsai method

As illustrated in Fig.8, an increase in nano clay content results in a higher elastic modulus, indicating that adding nano clay enhances the stiffness of the composite. This observation aligns with previous research showing improved mechanical properties in epoxy systems with greater filler content. However, while the three-phase Halpin-Tsai method offers a reasonable estimate, it does not account for filler agglomeration, which can detrimentally affect mechanical properties by creating stress concentration areas that reduce stiffness and strength. This limitation may clarify the discrepancies between experimental and theoretical results at higher filler concentrations, as discussed in several studies. This figure indicates that the Halpin-Tsai model predicts the mechanical properties of the composite effectively at lower nano clay levels, where dispersion is optimal. However, its accuracy declines with higher filler concentrations due to agglomeration risks, as noted by researchers like Khosla et al[10]. and Gupta & Mishra[27]. Improving processing techniques, such as enhanced dispersion methods, could potentially enhance model accuracy by mitigating agglomeration effects

3.7 Shear Test

As depicted in Fig. 9 and Table 4 , the initial addition of 0.5 wt.% CNT to the epoxy composite leads to a noticeable increase in shear modulus of approximately 6.85%. suggesting an enhancement in the material rigidity. This is

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consistent with the findings in previous studies (e.g., references [18, 24, 25]), which report improved mechanical properties, including shear strength and modulus, with the incorporation of CNT in polymer matrices due to their exceptional stiffness and high aspect ratio. Further addition of NC up to 5 wt.% into the epoxy/CNT nanocomposite results in continued increases in shear modulus and shear strength. This improvement can be attributed to the high surface area-to-volume ratio of nanofillers, which enhances interfacial bonding between the matrix and fillers, as discussed in references [10, 25, 26]. This increased interfacial area leads to a reinforced composite structure where stress is more effectively transferred from the matrix to the nanofillers, thereby improving the material's load-bearing capacity. However, the bar chart also reveals that beyond a 5 wt.% NC concentration, the shear modulus, and strength begin to decrease. This trend aligns with the limitations noted in previous research, such as by Khosla et al. [10] and Gupta & Mishra, where higher filler loadings can lead to agglomeration. Agglomeration disrupts the uniform dispersion of nanofillers, creating stress concentration points that weaken the composite’s mechanical properties. This decrease is a known challenge in nanocomposite fabrication and suggests that exceeding a critical filler concentration can compromise the expected reinforcement benefits.

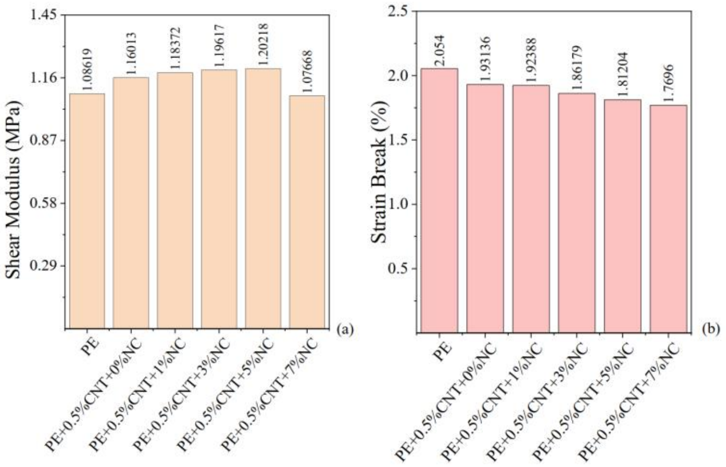

Fig. 9: (a)Shear Modulus and (b) Strain break

Table 4: The effect of NC on shear modulus of nanocomposite

	EP	EP-0.5%CNT	EP-0.5%CNT-1%NC	EP-0.5%CNT-3%NC	EP-0.5%CNT-5%NC	EP-0.5%CNT-7%NC
Shear Modulus (MPa)	1086.19	1160.13	1183.72	1196.17	1202.18	1076.68

- *Compare with the three-phase Halpin-Tsai method*

Fig. **10** presents a comparison between the experimentally obtained shear modulus and the theoretical predictions from the three-phase Halpin-Tsai method. Up to a nano clay (NC) concentration of approximately 3 wt.%, the Halpin-Tsai model closely estimates the experimental shear modulus, indicating good agreement. However, beyond this point, the experimental and theoretical lines diverge, with the experimental shear modulus showing a plateau or decline, while the theoretical prediction continues to increase. As indicated in previous studies (e.g., references [10, 26]), the Halpin-Tsai method does not account for the agglomeration or clustering of nanofillers, which becomes increasingly likely as the NC concentration rises. At higher filler content, nano clay particles tend to aggregate rather than disperse uniformly, leading to stress concentration zones within the composite matrix. These clusters disrupt the load transfer efficiency between the polymer and nanofillers, resulting in reduced stiffness and strength, which explains the decline observed in the experimental shear modulus. This behavior is consistent with findings from Khosla et al. [10] and Gupta & Mishra [27], which highlight that filler agglomeration negatively affects mechanical properties due to the non-uniform distribution of stress. Additionally, Ali et al. [26] and Nguyen et al. [21] discuss similar limitations in polymer composites, where the dispersion of nanofillers significantly influences the composite's effectiveness in enhancing mechanical properties.

3.8 Hot plate test

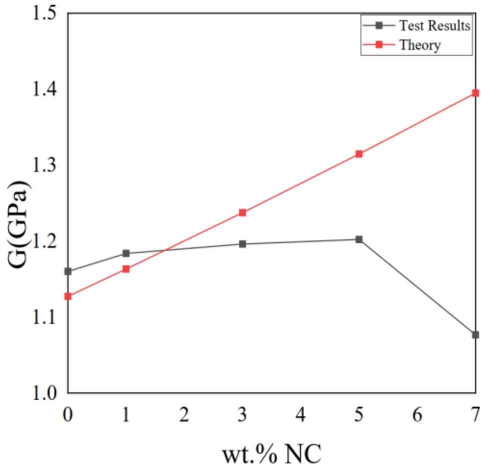

Fig. 10: Comparison of experimental shear modulus and theoretical results of the three-phase Halpin Tsai method

The hot plate test measures thermal conductivity by assessing heat flow through a sample placed on a heated plate. It's particularly useful for evaluating polymer-based nanocomposites, allowing researchers to observe how fillers like Carbon nanotubes (CNTs) and nanoclays (NC) affect thermal performance This test provides key insights into heat conduction, helping optimize material formulations for applications requiring efficient heat management. In Fig. 11(a), thermal conductivity variations of the pure epoxy and nanocomposite samples are plotted against temperature. Generally, the addition of nanoclay (NC) to epoxy/CNT nanocomposites does not consistently enhance thermal conductivity. Instead, it produces an irregular pattern, as shown by the fluctuations in thermal conductivity across different temperatures. This irregularity can be attributed to phonon scattering, a significant mechanism in thermal conduction within polymer nanocomposites. Phonon conduction is governed by lattice vibrations, but various scattering processes—such as acoustic mismatch at interfaces and imperfections at the matrix-nanofiller boundary—hinder phonon transport. These findings align with previous research, including Romano et al. [28] and Nguyen et al. [18], who identified that the presence of interfaces between the polymer matrix and nanofillers introduces flaws that lead to phonon scattering, thereby limiting thermal conductivity. In Fig. 11(b), the bar chart compares the thermal conductivity of epoxy reinforced solely with CNTs versus epoxy containing both CNTs and NC. The results indicate that the composite with both CNT and NC has lower thermal conductivity than the sample with only CNTs. Romano et al. [28] observed a similar trend, where the addition of NC alone did not improve the thermal conductivity of epoxy, suggesting that NC has limited effectiveness in enhancing thermal transport. While NC can improve the dispersion of CNTs within the matrix, it simultaneously introduces additional interfaces and potential aggregations that disrupt the CNT network, reducing the thermal pathways. This aggregation and bundling of CNTs in the presence of NC can weaken the continuity of CNT-CNT connections, which are crucial for thermal conduction. The decreased thermal conductivity is consistent with the findings that highlight the need for direct CNT-CNT linkages for efficient phonon transport, which NC appears to disrupt. Overall, the hot plate test results demonstrate that although the addition of NC can aid in the dispersion of CNTs, it ultimately reduces thermal conductivity in CNT-filled epoxy composites by disrupting CNT-CNT thermal pathways.

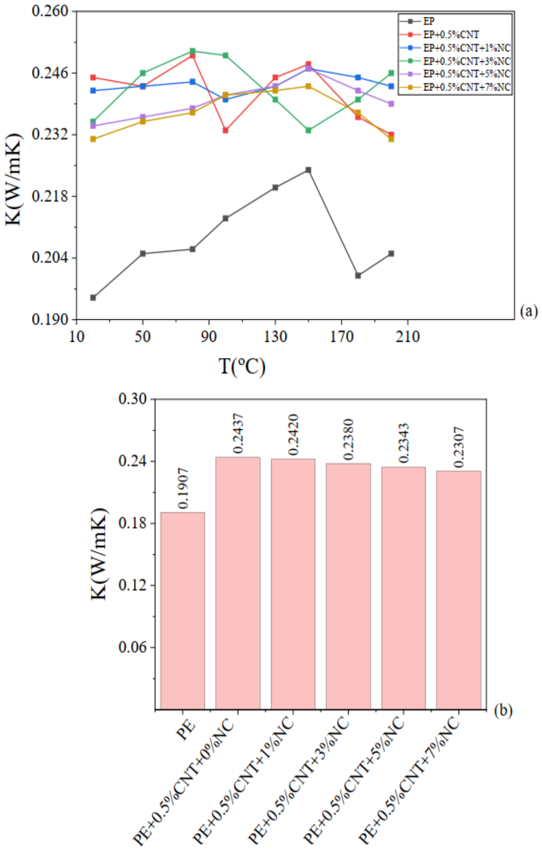

Fig. 11: The variations of thermal conductivity of the pure epoxy and nanocomposite samples (a) versus temperature, and (b) at 25˚C

3.9 Modal test

Modal testing is a technique employed to obtain the modal parameters of a structure. The Frequency Response Function (FRF), derived from this testing, provides critical information regarding fundamental frequencies, damping characteristics, and mode shapes. Typically, two excitation methods are utilized: impact hammer excitation and shaker excitation. In this study, the impact hammer excitation technique has been selected. While this method is relatively straightforward to implement, achieving consistent results can be challenging. Nevertheless, the primary advantage of this approach lies in its simplicity and minimal hardware requirements, resulting in shorter measurement times. The implementation of the impulse, illustrated in Fig.12, can be achieved using a hammer, an electromechanical

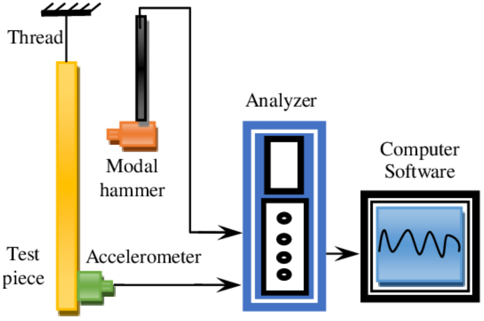

Fig.12: The schematic of the set-up for the impact hammer test

The modal testing was performed on a hybrid polymer nanocomposite (HPNC) structure with clamped-free (C-F) boundary conditions to assess the first three natural frequencies across various formulations (Table 5). As established, natural frequencies are influenced by material properties and boundary conditions, with frequency depending on both the stiffness and mass of the structure [1, 2]. Typically, increasing stiffness raises natural frequencies, whereas additional mass reduces them. The results indicate that the HPNC sample with 5wt.% nano clay (NC) and 0.5wt.% carbon nanotubes (CNT) achieved the highest frequencies across all modes tested, demonstrating the optimal enhancement of stiffness for this configuration. The observed trend of increasing NC content correlating with higher frequencies aligns with findings in the literature. For instance, Ayatollahi et al. [4] and Wang et al. [5] report that NC and CNT improve the composite stiffness, resulting in higher vibrational performance. Similarly, Choi and Ryu [3] observed that NC and CNT additions increase the elastic modulus in epoxy nanocomposites, which supports higher natural frequencies. These findings are consistent with prior research indicating that NC and CNT additions increase stiffness while maintaining manageable mass, thus improving the composite frequency response, as shown by Sinha Ray [29] and Liu and Grunlan [1, 2]. Zhang et al. [23,25] and Gupta and Mishra [27] also exhibited synergistic enhancements in stiffness and frequency performance with NC and CNT inclusions. Overall, these results indicate that moderate NC and CNT concentrations offer a favorable balance of stiffness and mass, leading to improved natural frequencies. This combination of 5wt.% NC and 0.5wt.% CNT is thus optimal for maximizing vibrational performance in hybrid nanocomposites, consistent with findings by Nguyen et al. [9], Maurya et al. [11], and Dhaliwal et al. [30].

Table 5 the first three frequencies for C- F boundary condition

Samples	f1 (Hz)	f2 (Hz)	f3 (Hz)
EP	142	386	637
EP-0.5%CNT	152	431	733
EP-0.5CNT-1%NC	153	438	741
EP-0.5%CNT-3%NC	155	447	764
EP-0.5%CNT-5%NC	203	546	968
EP-0.5%CNT-7%NC	160	460	813

Table 6 the first three frequencies for the C- F boundary condition based on the- phase Halpin- Tsai

Samples	f1 (Hz)	f2 (Hz)	f3 (Hz)

EP	132.95	833.22	2333.27
EP-0.5%CNT	133.17	834.63	2337.22
EP-0.5CNT-1%NC	133.46	86.41	2342.22
EP-0.5%CNT-3%NC	134.03	840.03	2352.34
EP-0.5%CNT-5%NC	134.62	843.71	2362.64
EP-0.5%CNT-7%NC	135.22	847.44	2373.10

Table 6 illustrates three frequency modes (f1, f2, f3) for different sample formulations, allowing for a visual comparison between the experimental results and the Halpin-Tsai predictions. The differences between the experimental and theoretical fundamental frequency results can be attributed to several factors. Firstly, the Halpin-Tsai method relies on idealized assumptions regarding the uniform dispersion and perfect interaction of nanofillers within the epoxy matrix, which may not accurately reflect the complexities and imperfections present in real materials material heterogeneity, such as variations in filler distribution and the presence of defects which can lead to a reduction in stiffness and, consequently, lower observed frequencies. Additionally, real materials exhibit viscoelastic behavior, introducing damping effects that are not accounted for in theoretical calculations. The synergistic or antagonistic interactions between carbon nanotubes and nano clay can also influence the mechanical properties beyond what is predicted theoretically. Finally, external factors, including environmental conditions like temperature and humidity, can further impact the mechanical response of the composites, contributing to the discrepancies observed between the two sets of results.

4. Conclusions

This study demonstrates that incorporating carbon nanotubes (CNTs) and nanoclay (NC) into the LY 5052 epoxy matrix significantly enhances its thermal and mechanical properties, making it a viable material for advanced engineering applications. Optimal improvements in properties such as thermal stability, glass transition temperature, modulus, and strength were achieved at 0.5 wt% CNTs and 5 wt% NC, consistent with findings in similar research [3, 6, 23] where hybrid fillers significantly enhanced mechanical strength and thermal stability. The three-phase Halpin-Tsai model accurately predicted mechanical properties that correlated closely with experimental results, though slight deviations were noted, which aligns with previous studies that observed similar minor variations due to assumptions of ideal dispersion [7, 10]. These discrepancies likely stem from the theoretical model assumptions, such as ideal dispersion and bonding between fillers and matrix, which are difficult to replicate experimentally. Practical factors, including minor agglomeration, imperfect filler distribution, and potential interactions between CNT and NC surfaces, contribute to variations from theoretical predictions. While 5% NC was found to enhance CNT dispersion and improve composite strength, higher NC levels led to clustering, and diminishing performance. This research shows that a balanced combination of CNTs and NC can overcome dispersion challenges and optimize composite properties, supporting its use in aerospace, automotive, and electronics applications. Future research directions could explore alternative methods to improve CNT dispersion further, such as surface functionalization techniques or hybrid surfactants. Additionally, investigating the effects of various nanoclays or other nanofillers (e.g., graphene oxide, metal oxides) on composite properties may yield insights into even higher-performance nanocomposites. Finally, refining predictive models to account for non-ideal dispersion and complex filler interactions could enhance the accuracy of theoretical predictions and aid in designing next-generation materials.

Nomenclature

All variables used in this manuscript are listed in nomenclature.

NC	Nano Clay
CNT	Carbon Nano Tube
MWCNT	Multi-Walled Carbon Nano Tube
HPNC	Hybrid polymer nanocomposites
ϕ_f	The volume fraction of nanofiller
M	Mechanical feature
α	Shape Factor
f	indice f present filler
m	Indice m present matrix
E’	Storage Modulus

υ_e	Cross-linking density
T_g	Glass Transition Temperature

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Conflicts of Interest

The author declares that there is no conflict of interest regarding the publication of this article.

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