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Sustainable Epoxy Composites Enhanced by Cellulose Nanocrystals for Structural and Thermal Applications

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Abstract

This study presents the advancement and behaviour analysis of sustainable nanocomposites reinforced with cellulose nanocrystals (CNCs), a biodegradable and high-aspect-ratio nanofiller derived from microcrystalline cellulose. CNCs were incorporated into a bisphenol-A-based epoxy matrix at varying concentrations (0, 1, 3, 5, and 7 wt%) to assess their influence on mechanical, thermal, and viscoelastic properties. The tensile strength of the composites increased significantly, from 45 MPa for neat epoxy to 65 MPa at 5 wt% CNC, while Young's modulus showed an improvement from 1.8 GPa to 3.0 GPa. Thermogravimetric analysis revealed an enhancement in thermal behaviourthat is attributed to the barrier effect of CNC-derived char layers. Dynamic mechanical analysis showed a rise in glass transition (T_g) from 118 °C to 124 °C and a 50% increase in storage modulus, indicating reduced chain mobility and enhanced stiffness under dynamic loading. However, a marginal drop in all properties was observed at 7 wt% CNC due to particle agglomeration and interfacial inefficiencies. These findings suggest that CNCs can be effectively utilized as green reinforcements in thermosetting polymers, with 5 wt% CNC loading offering the most favorable balance between mechanical strength, thermal resistance, and processing feasibility, making them promising candidates for lightweight, eco-friendly applications in aerospace, automotive, and electronic sectors.

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INTRODUCTION

The 21st century has witnessed a paradigm shift in materials science, where sustainability, biodegradability, and eco-efficiency have become critical design drivers. Traditional structural and functional materials—especially petroleum-derived polymers and composites—though high-performing, pose significant environmental concerns due to their non-renewable origin, carbon footprint, and end-of-life disposal issues [1]. In response, researchers are increasingly turning to renewable, biodegradable, and carbon-neutral resources to develop next-generation composite materials that balance performance with environmental stewardship.

Among emerging green reinforcements, cellulose nanocrystals (CNCs) have garnered significant

interest as a class of high-aspect-ratio, bio-based nanofillers that can rival the mechanical performance of synthetic nanomaterials while remaining entirely biodegradable and non-toxic [2–4]. CNCs are rod-like crystalline domains extracted from natural cellulose, the most abundant biopolymer on Earth, typically derived from wood pulp, cotton, flax, or agricultural residues [5]. The extraction of CNCs typically involves controlled acid hydrolysis of lignocellulosic biomass, which removes amorphous regions, leaving behind a highly ordered crystalline structure [6–8].

CNCs possess a high surface area (200–400 m^2/g) and are functionalized with abundant hydroxyl groups and even chemical grafting with compatible polymer matrices [9,10]. Their nanoscale dimensions also enable them to reinforce the polymer at the molecular level, improving modulus, tensile strength, dimensional stability, thermal resistance, and in some cases, barrier properties, all at low filler loadings (<5 wt%) [11–13].

Epoxy resins, in particular, present a compelling matrix for CNC reinforcement due to their widespread industrial use, tunable crosslinking chemistry, and inherently high stiffness and chemical resistance [14,15]. However, neat epoxy is brittle and lacks the flexibility and toughness required for certain advanced applications, such as impact-resistant aerospace laminates or crash-safe automotive components [16]. Incorporating CNCs into epoxy matrices provides an opportunity to tailor these properties while simultaneously improving environmental sustainability.

Prior research has shown that the incorporation of CNCs into epoxy systems can enhance performance metrics across several dimensions. Fortunati et al. [17] demonstrated that CNCs improve epoxy's modulus and thermal stability, while Liu et al. [18] found that CNC-reinforced epoxies exhibited better shape stability at elevated temperatures. Moreover, CNCs can serve as nucleating agents, facilitating improved resin curing uniformity and reducing microvoid formation during crosslinking [19]. Their surface chemistry also allows for chemical functionalization, such as silanization or grafting with reactive epoxide or amine groups, to improve interfacial bonding and dispersion in non-polar epoxy matrices [20,21].

Despite their advantages, several challenges of CNCs in epoxy systems include:

- Agglomeration of CNCs due to strong hydrogen bonding and van der Waals interactions, especially at higher loadings.
- Incompatibility with hydrophobic polymer matrices without surface treatment or use of coupling agents.
- Reduction in ductility and increase in brittleness at excessive nanofiller concentrations.
- Difficulties in uniform dispersion, which can lead to phase separation, voids, and interfacial failure under mechanical stress [22–24].

Therefore, it is essential to optimize CNC content, employ appropriate dispersion techniques (e.g., ultrasonication, solvent blending, high-shear mixing), and characterize the interfacial interaction mechanisms to realize the full potential of CNC/epoxy nanocomposites.

Furthermore, as the global focus intensifies on green composites for sectors such as aerospace, automotive, marine, packaging, and electronics, CNC-epoxy systems emerge as promising contenders due to their synergy of performance and environmental responsibility. Their use can contribute to lightweighting strategies, reduce reliance on synthetic fillers like carbon fibers or glass fibers, and offer end-of-life advantages, such as compostability or thermal recovery [25,26].

This study aims to develop, characterize, and optimize cellulose nanocrystal-reinforced epoxy composites to achieve high-performance materials that are not only strong and thermally stable but also sustainable and lightweight. CNCs were extracted from microcrystalline cellulose and incorporated into a bisphenol-A-based epoxy matrix at loadings ranging from 1–7 wt%. A range of experimental techniques, including tensile testing, thermogravimetric analysis (TGA)were used to evaluate the effect of CNCs on the structural, and mechanical performance of the composites.

Through this investigation, we seek to identify the optimal filler loading that maximizes mechanical performance and thermal resistance while minimizing processing challenges and property trade-offs. The insights from this study will contribute to the broader goal of transitioning towards sustainable nanocomposite technologies, suitable for the next generation of lightweight, multifunctional materials.

MATERIALS AND METHODS

Cellulose nanocrystals (CNCs) were obtained from microcrystalline cellulose (MCC) a derivative from wood. Analytical-grade sulfuric acid (H_2SO_4, 64%) was procured and used for hydrolysis without further purification. Epoxy resin and a polyamine hardener were obtained from a commercial supplier and used in the manufacturer-recommended stoichiometric ratio. Ethanol (95%) was used as a dispersion medium for CNCs prior to composite fabrication. All reagents and chemicals employed in this study were of analytical grade.

For CNC extraction, MCC was hydrolyzed by slow addition into 64% H_2SO_4 under vigorous stirring at 45 °C for 45 minutes. The reaction was quenched using cold deionized water. The solution was dialyzed in deionized water for 5–7 days and then sonicated using a probe sonicator at 200 W for 10 minutes to disperse agglomerates. The resulting stable CNC colloid was freeze-dried for 48 hours to obtain CNC powder.

To fabricate the CNC-reinforced epoxy composites, CNCs were dispersed in ethanol and ultrasonicated for 30 minutes to ensure de-agglomeration. The dispersed CNCs were slowly introduced into the epoxy resin and mechanically stirred at 500 rpm for 30 minutes. The resulting stable CNC colloid was freeze-dried for 48 hours to obtain CNC powder, ensuring complete removal of moisture. Prior to composite fabrication, CNCs were re-dispersed in ethanol and ultrasonicated, and ethanol was allowed to evaporate under vacuum at 50 °C for 12 hours to eliminate residual solvent and avoid interference during curing.

Hardener was then added and mixed thoroughly using a planetary centrifugal mixer for 5 minutes under vacuum to avoid bubble formation. The resulting blend was poured into silicone molds to achieve flat sheet specimens of dimensions 120 mm × 120 mm × 3 mm. The CNC loading in the epoxy matrix was varied at 0, 1, 3, 5, and 7 wt% to evaluate its effect on material performance.

Prepared specimens were conditioned at 25 °C, 50% relative humidity for 24 hours prior to testing. Mechanical testing was conducted using a universal testing machine (Instron 3369) per ASTM D638 Type V for tensile properties. Mechanical testing was conducted using a universal testing machine (Instron 3369) per ASTM D638 Type V for tensile properties, with five replicates (n=5) tested for each CNC concentration to ensure statistical relevance. DMA was carried out on rectangular test samples (60 mm × 10 mm × 2 mm), scanned from 25 to 150 °C. Dynamic mechanical analysis (DMA) was performed on three specimens per condition (n=3), scanned from 25 to 150 °C.

Thermal stability was assessed using thermogravimetric analysis (TGA) under nitrogen atmosphere from 30–600 °C at 10 °C/min. FTIR was carried out to monitor functional group interactions between CNCs and the epoxy matrix.

RESULTS AND DISCUSSION

Mechanical Behaviour

The tensile strength of CNC-epoxy composites increased with CNC loading up to 5 wt%, peaking at 65 MPa, representing a 44% improvement over neat epoxy (45 MPa), as shown in Figure 1.

This enhancement is attributed to the excellent load transfer capability of well-dispersed CNCs due to their high aspect ratio and modulus (~100–150 GPa). CNCs act as stress concentrators and efficiently transfer load across the matrix, provided that the dispersion and interfacial adhesion are optimized [27,28]. The mechanical reinforcement also correlates with a 66% increase in Young's modulus (Figure 2).

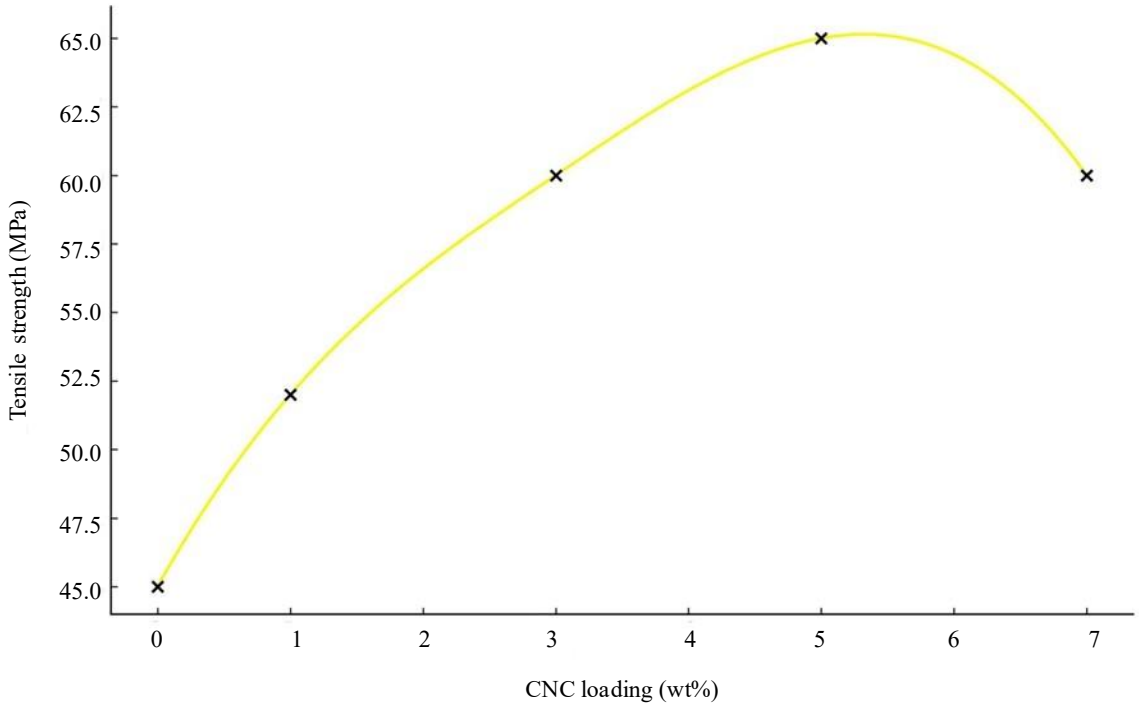

Figure 1. Tensile strength vs CNC loading.

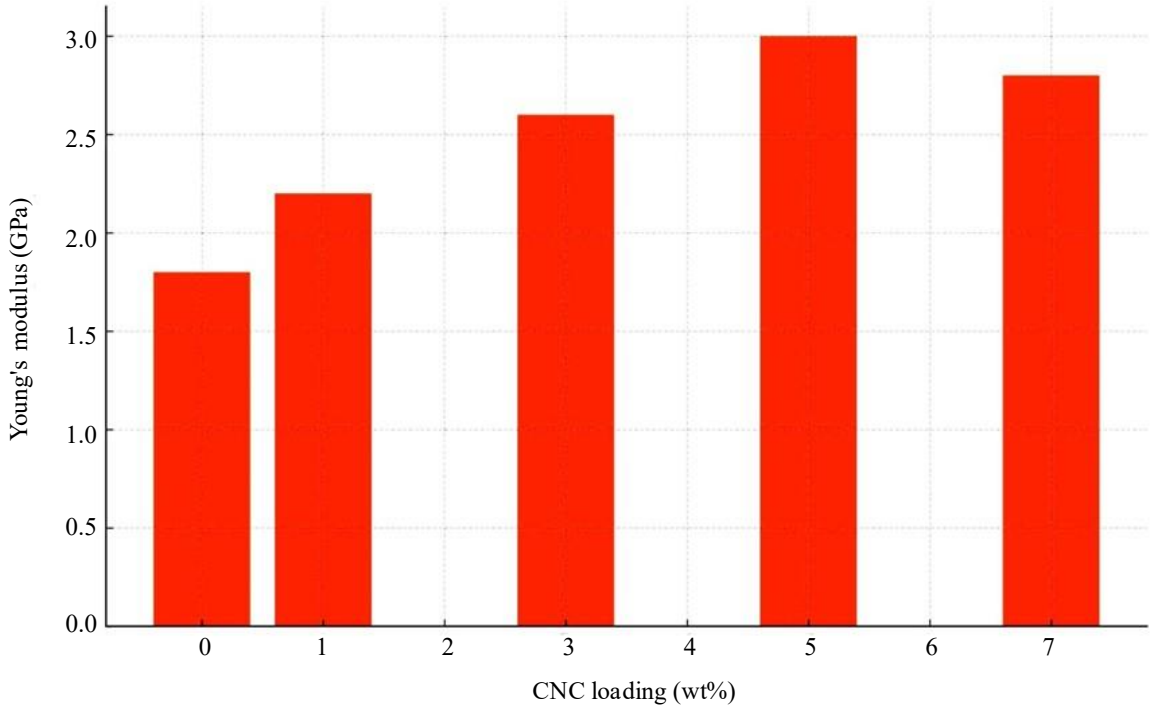

Figure 2. Young's modulus vs CNC loading.

However, at 7 wt%, both tensile strength and modulus dropped slightly to 60 MPa and 2.8 GPa, respectively. This decline is likely due to CNC agglomeration, which can introduce voids and stress-concentration sites that act as crack initiators during tensile loading [29]. Studies have shown that beyond a critical loading, the CNCs' tendency to hydrogen bond among themselves leads to microcluster formation, disrupting matrix continuity [30]. Furthermore, excess CNCs can restrict resin mobility during curing, potentially reducing crosslinking efficiency and thus tensile properties [31].

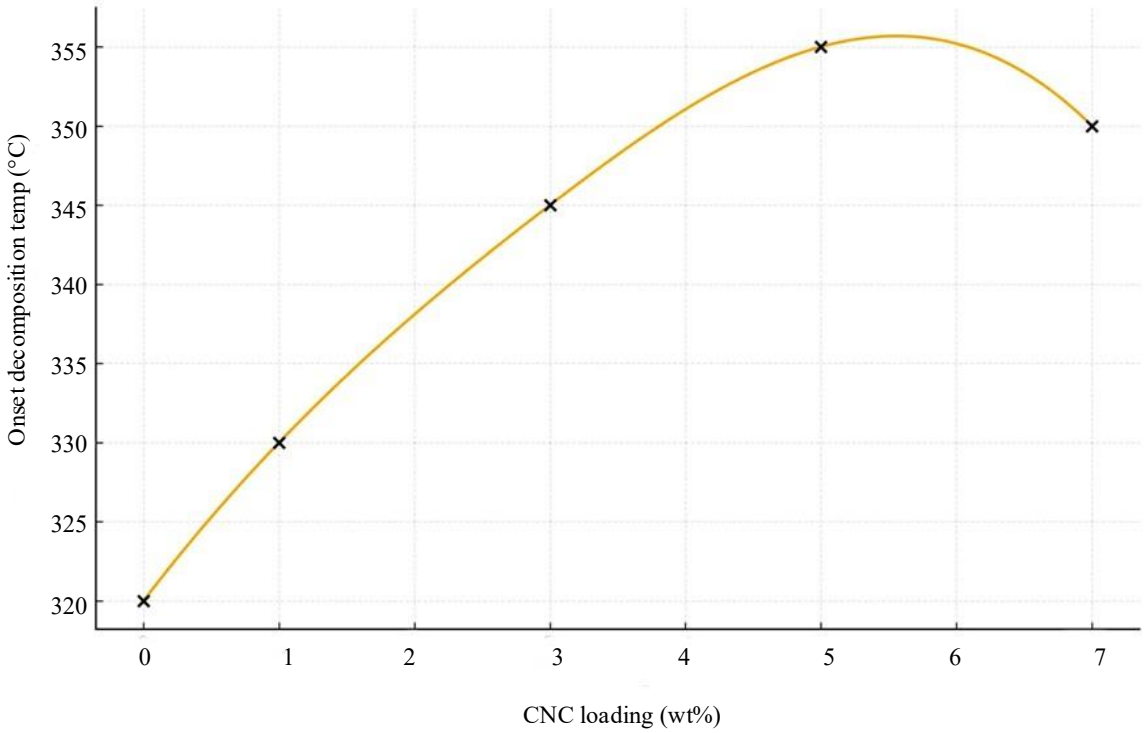

Figure 3. Thermal stability (onset decomposition temperature) vs CNC loading.

Thermal Stability

Thermal stability analysis of CNC-epoxy composites revealed thatthe onset decomposition temperature (Td) increased with CNC content; rising from 320 °C (neat epoxy) to 355 °C at 5 wt% CNC (Figure 3).

This 35 °C enhancement suggests that CNCs act as thermal insulators, delaying the heat-induced breakdown of polymer chains. The surface of CNCs forms char residues under heating, which can act as a barrier to oxygen diffusion and heat transfer, thus improving the thermal decomposition threshold [32,33].

At 7 wt%, the onset temperature slightly decreased to 350 °C, which is still higher than neat epoxy but reflects diminishing returns. Excess CNCs may form aggregates that reduce thermal conductivity uniformity or even catalyze localized degradation due to unprotected zones within the matrix [34]. Similar non-linear improvements in Td with nanofillers have been reported in other studies on starch- and PLA-based nanocomposites [35].

Glass Transition Temperature (Tg)

DMA revealed an increase in the T_g from 118 °C for epoxy to 124 °C for the 5 wt% CNC composite (Figure 4).

The increase in Tg signifies a reduction in polymer chain mobility, attributed to the physical confinement introduced by CNCs and the strong CNC–epoxy interfacial bonding [36]. As CNCs are embedded within the matrix, they restrict the movement of polymer segments, requiring more energy (i.e., higher temperature) to initiate the glass transition.

However, a slight decrease in Tg was observed at 7 wt% (123 °C), indicating that excessive CNCs may lead to phase discontinuities and reduce network uniformity [37]. Moreover, interactions among CNC clusters may prevent proper filler–matrix contact, undermining the reinforcing effects [38].

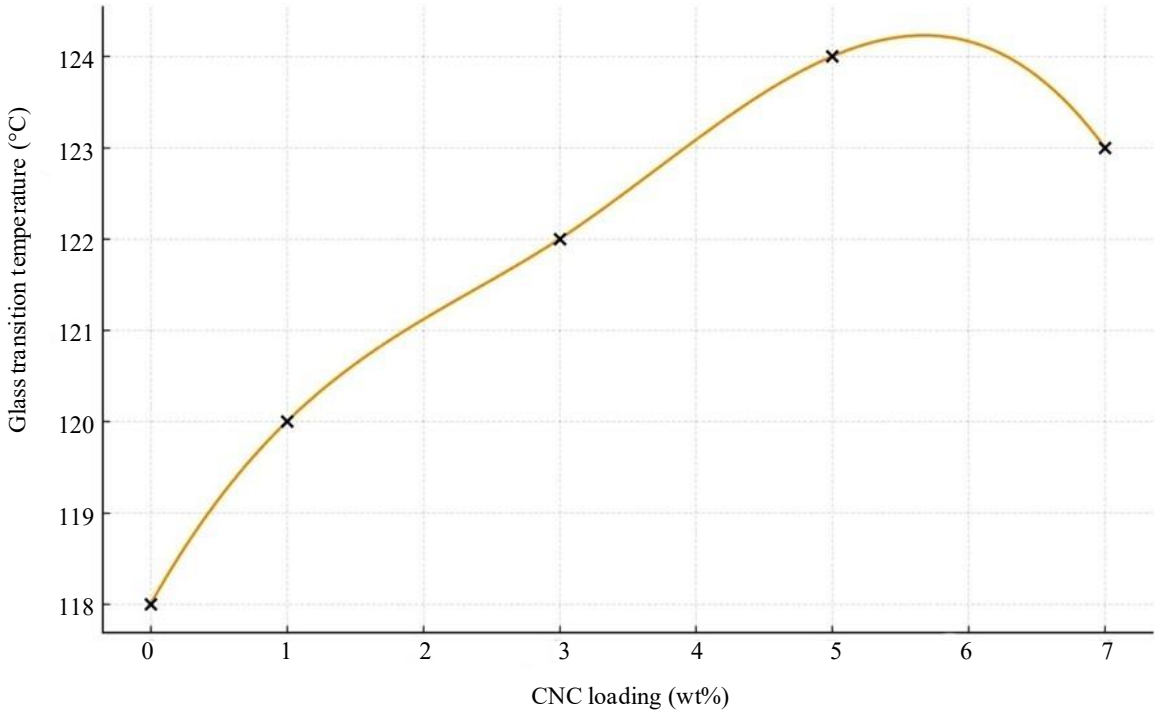

Figure 4. Glass transition temperature (Tg) vs CNC loading.

Viscoelastic Behaviour: Storage Modulus

The storage modulus (E′), which quantifies material stiffness under oscillating stress, increased substantially with CNC content (Figure 5). The storage modulus at room temperature rose from ~1000 MPa to 1500 MPa with 5 wt% CNC, a 50% enhancement. This confirms that CNCs serve as effective nanoscale reinforcements, providing dimensional and elastic stability under dynamic loads [39].

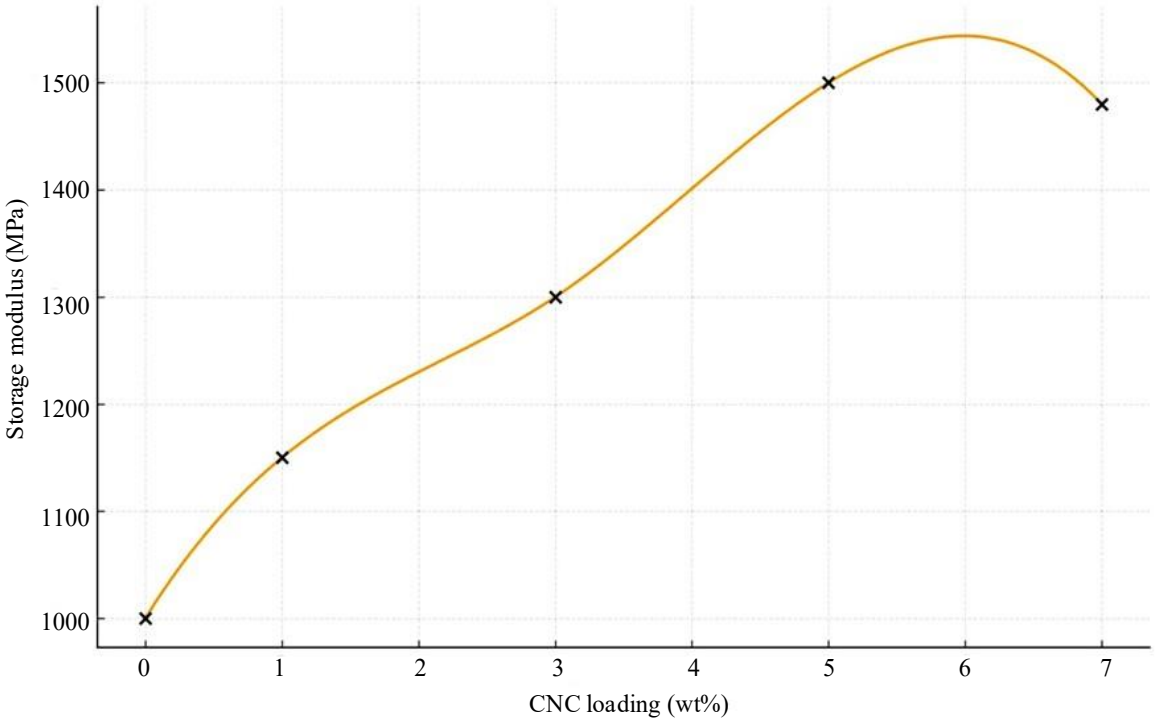

Figure 5. Storage modulus vs CNC loading.

The improvement in E′ is attributed to the rigid crystalline domains of CNCs anchoring within the crosslinked epoxy network, reducing the energy loss through viscoelastic deformation. These findings are consistent with prior reports where CNC-reinforced biopolymers demonstrated marked increases in both stiffness and temperature-dependent modulus values [40]. Beyond 5 wt%, however, the increase in E′ becomes marginal due to aggregation effects, consistent with the mechanical property trends.

Overall Performance Analysis

The synergy between mechanical, thermal, and viscoelastic properties suggests that 5 wt% CNC loading represents an optimum balance for property enhancement. At this concentration, CNCs are well-dispersed, maintain strong interfacial adhesion, and reinforce the epoxy without causing agglomeration or processing difficulties. The observed trends in performance metrics align with literature on nano-reinforced epoxies using fillers [41-45].

CONCLUSION

The present study explored the influence of CNC loading on the properties of epoxy composites. The insights derived offer guidance for the rational design of bio-based nanocomposites suited for high-performance applications.

Based on experimental data, the following conclusions are drawn:

- *Enhanced mechanical properties:* The addition of 5 wt% CNC led to a peak tensile strength of 65 MPa and Young's modulus of 3.0 GPa, showing a 44% and 66% enhancement respectively over neat epoxy.
- *Thermal stability improvement:* Thermal stability improved from 320 °C to 355 °C due to their char-forming behaviour.
- *Elevated glass transition temperature:* The Tg increased from 118 °C (neat epoxy) to 124 °C at 5 wt%, indicating better resistance and restricted chain mobility.
- *Superior viscoelastic performance:* Storage modulus improved by 50% with 5 wt% CNC, confirming improved stiffness under dynamic conditions.
- *Optimal filler loading identified:* Beyond 5 wt%, performance declined slightly due to CNC agglomeration, suggesting that 5 wt% is the ideal reinforcement level for property optimization.

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