Scientific Research Publishing

Open Journal of Applied Sciences, 2025, 15(7), 2091-2100
https://www.scirp.org/journal/ojapps
ISSN Online: 2165-3925
ISSN Print: 2165-3917

Enhancing Thermo-Mechanical Properties of Epoxy Nanocomposites with Dispersed MWCNTs Using a Novel Hybrid Mixing Technique

Akhilesh Singh[1*], Somveer Singh[1], Anurag Mishra[1], Ashutosh Singh[1], Abhishek Singh[2], Anil Kumar Soni[3]

[1]Department of Chemistry, K.S. Saket P.G. College, Ayodhya, India
[2]Department of Chemistry, U.P. Autonomous College, Varanasi, India
[3]Department of Chemistry, Shia P.G. College, Lucknow, India
Email: akhileshchem@gmail.com, bsomveersingh@gmail.com, anuragm878@gmail.com, asinghkssaket@gmail.com, abhupc@gmail.com, anilsony82@gmail.com

How to cite this paper: Singh, A., Singh, S., Mishra, A., Singh, A., Singh, A. and Soni, A.K. (2025) Enhancing Thermo-Mechanical Properties of Epoxy Nanocomposites with Dispersed MWCNTs Using a Novel Hybrid Mixing Technique. *Open Journal of Applied Sciences*, **15**, 2091-2100.
https://doi.org/10.4236/ojapps.2025.157137

Received: June 3, 2025
Accepted: July 18, 2025
Published: July 21, 2025

Abstract

This study presents an innovative approach to enhance the thermo-mechanical properties of multi-walled carbon nanotube (MWCNT) reinforced epoxy nanocomposites through a dual-action dispersion method. By simultaneously applying ultrasonic energy and shear force via an axial-flow impeller, a uniform distribution of MWCNTs within the epoxy matrix was achieved without the need for chemical modification. The impact of MWCNT loading (0.25 - 1.0 wt%) on the thermal and mechanical behavior of the composites was systematically investigated. Dynamic mechanical analysis revealed a significant improvement in storage modulus, with a maximum enhancement observed at 0.75 wt% MWCNT content. Thermogravimetric analysis demonstrated increased thermal stability, confirming the efficiency of nanotube reinforcement. Scanning electron microscopy of fracture surfaces validated the homogeneity of nanotube distribution and provided insights into fracture mechanics. These findings suggest that the adopted hybrid dispersion technique is effective for fabricating high-performance polymer nanocomposites for structural applications.

Keywords

MWCNTs, Epoxy Resin and Nanocomposites

1. Introduction

Carbon nanotubes (CNTs), particularly multi-walled carbon nanotubes (MWCNTs), have garnered extensive attention since their discovery by Iijima in 1991 due to their extraordinary mechanical strength, electrical conductivity, and thermal stability [1]. These characteristics make MWCNTs exceptional candidates for reinforcing polymers, ceramics, and metals across a wide range of applications, including aerospace, automotive, and electronics [2] [3].

In polymer nanocomposites, the integration of MWCNTs has been shown to significantly improve mechanical stiffness, toughness, and thermal performance [4]-[6]. However, the full potential of MWCNTs can only be realized if they are uniformly distributed and strongly bonded within the polymer matrix. Aggregation of nanotubes—caused by van der Waals forces and high surface energy—often results in inhomogeneous composites with reduced or inconsistent performance [7] [8].

To address the issue of dispersion, researchers have explored various strategies, such as mechanical stirring, ultrasonication, melt blending, and chemical functionalization of the nanotube surfaces [9]-[11]. While chemical functionalization can improve dispersibility and interfacial bonding, it may also compromise the intrinsic structure and properties of CNTs [12]. Thus, physical dispersion methods remain a desirable alternative for preserving nanotube integrity while achieving effective distribution. Previous studies have shown that combining ultrasonication with shear mixing can significantly improve dispersion quality [13] [14]. For instance, Thostenson *et al.* demonstrated the effectiveness of high shear extrusion in dispersing CNTs in thermoplastics [15], while Guadagno *et al.* reported improvements in epoxy composites using ultrasonication [16]. Nevertheless, there remains a need for scalable, efficient techniques that ensure both uniform dispersion and enhanced interaction with the matrix.

The enhanced thermo-mechanical properties achieved through uniform dispersion of MWCNTs make these nanocomposites promising candidates for structural and functional components in high-performance applications. Specifically, they can be applied in aerospace parts where weight-to-strength ratio is critical, automotive components requiring impact resistance and durability, and electronic encapsulations where thermal stability and conductivity are vital. Such applications justify the need to optimize dispersion techniques to achieve superior mechanical and thermal performance in the final composite. In this work, we propose a novel hybrid dispersion technique that simultaneously employs ultrasonic waves and mechanical shear force using an axial-flow impeller. This synergistic approach aims to de-agglomerate MWCNTs effectively and distribute them uniformly throughout an epoxy resin. The focus of this study is to investigate how this improved dispersion influences the thermo-mechanical properties of the resulting nanocomposites, including storage modulus, glass transition temperature, and thermal stability.

2. Experimental Procedure

2.1. Materials

The primary components for composite fabrication included multi-walled carbon nanotubes (MWCNTs) produced via chemical vapor deposition (CVD), with an average diameter of approximately 30 nm and the length of MWCNTs is fall in the range of 20 to 45 μm. The cobalt oxide nano particles on alumina substrate are used as catalyst in growth of MWCNTs. The polymer matrix was an epoxy resin (Camcoat-2071 Bisphenol A based) paired with a compatible aliphatic hardener (HY-951 amine based), both sourced from Champion Advanced Materials Pvt. Ltd., India.

2.2. Composite Fabrication Procedure

A predetermined amount of MWCNTs (ranging from 0.25 to 1.0 wt%) was first dispersed into the epoxy resin with the aid of 10% acetone to temporarily reduce viscosity and assist in preliminary mixing. This mixture underwent manual stirring with a glass rod to pre-disperse the nanotubes.

Subsequently, a hybrid dispersion process was employed. The MWCNT-epoxy-acetone mixture was treated using a dual-action technique: ultrasonic energy was applied using a titanium alloy (Ti-6Al-4V) probe (13 mm diameter) operating at 20 kHz and 750 W, while simultaneously agitating the mixture using an axial-flow impeller (Mixture volume-50 ml, Impeller blade angle-45˚, Diameter of impeller-2.5 cm). The probe was positioned outside the primary vortex zone to enhance cavitation and disrupt MWCNT clusters through repeated sonic pulses. The impeller, rotating at 400 rpm, ensured effective flow dynamics and shear force throughout the 50 mL batch. The sonication amplitude was set at 60% with intermittent pulsing (10 s on/10 s off) for a duration of 30 minutes. To maintain thermal stability during dispersion, the beaker was externally cooled, limiting the temperature rise to 45 ˚C.

After processing, the acetone was allowed to evaporate completely. The hardener (10 wt% of total epoxy weight) was then thoroughly blended into the MWCNT-epoxy mixture. The resultant formulation was degassed under vacuum to eliminate entrapped air bubbles and poured into silicone rubber molds to shape the samples. Curing was performed in a hot air oven at 50˚C for 12 hours.

2.3. Dynamic Mechanical Analysis (DMA)

Thermo-mechanical behavior was analyzed using a PerkinElmer DMA 8000 system configured in single cantilever bending mode. Tests were conducted in the temperature range of 35˚C to 160˚C, with a controlled heating rate of 2˚C/min and a constant frequency of 1 Hz, following ASTM D4065 guidelines. The sample dimensions were approximately 9.2 mm × 7.5 mm × 2.5 mm.

2.4. Thermogravimetric Analysis (TGA)

Thermal stability was assessed using a STA-204 F1 instrument from Netzsch. Random fragments from cured composite samples were heated from room temperature to 550˚C at a rate of 10˚C/min under nitrogen flow. Alumina served as the reference material.

2.5. Morphological Characterization

To evaluate the dispersion of MWCNTs and the fracture surface features, field emission scanning electron microscopy (FESEM) was performed on gold-coated tensile fracture samples. An accelerating voltage of 15 kV was applied during imaging.

3. Results and Discussion

3.1. Thermo-Mechanical Behavior

The dynamic mechanical performance of the MWCNT-reinforced epoxy composites was evaluated to determine the influence of nanotube content and dispersion quality on stiffness and thermal transitions. As shown in **Figure 1**, the storage modulus (E') of the nanocomposites exhibited a noticeable increase with increasing MWCNT content up to 0.75 wt%, beyond which the modulus slightly declined. At 35˚C, a 0.75 wt% loading led to an approximately 35% improvement in modulus compared to the neat epoxy, suggesting efficient stress transfer through a well-dispersed nanotube network.

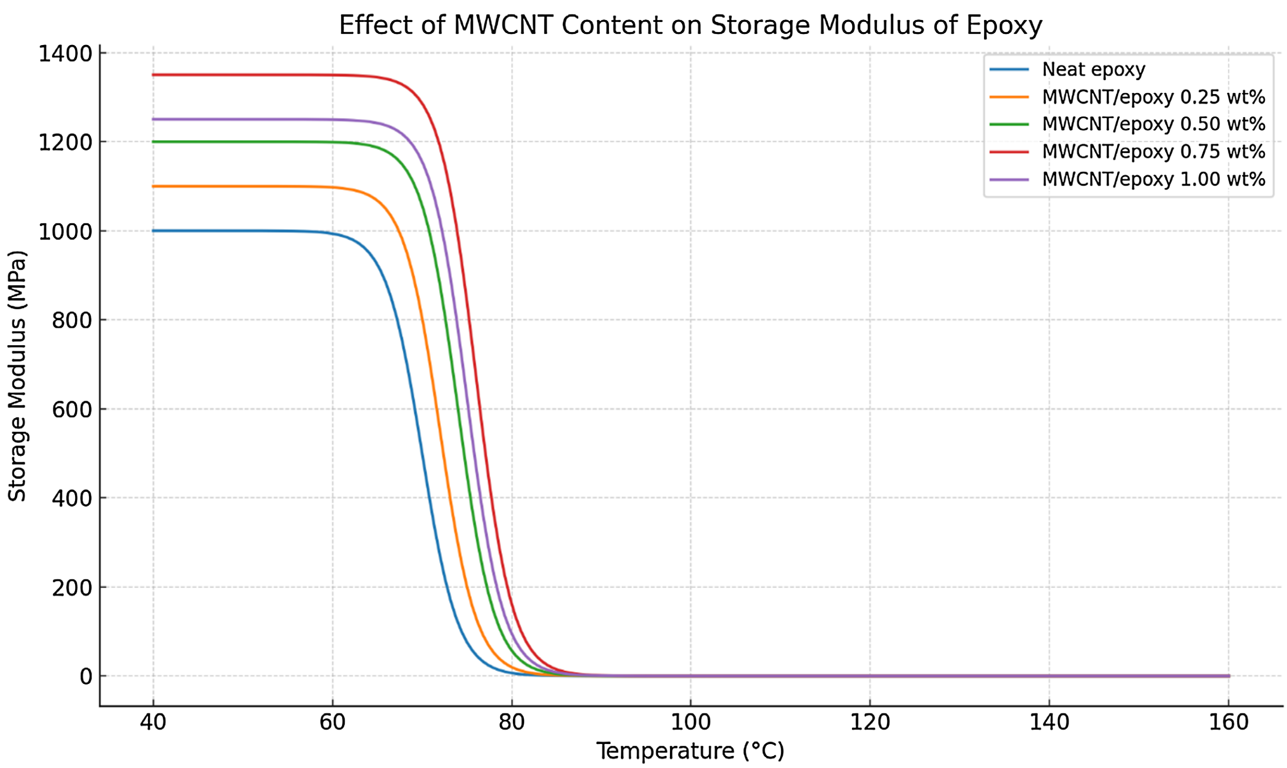

Figure 1. Storage modulus (E′) as a function of temperature for neat epoxy and MWCNT/epoxy nanocomposites. A significant increase in modulus is observed up to 0.75 wt% MWCNT loading, indicating improved stiffness due to enhanced dispersion and interaction.

The reduction in modulus at 1.0 wt% MWCNT may be attributed to the onset of nanotube aggregation, which disrupts the continuous polymer network and diminishes reinforcement efficiency. Similar non-linear trends in modulus with increasing filler content have been reported in earlier studies [17] [18].

The glass transition temperature (Tg), as determined from the peak of the tan δ curves (**Figure 2**), also shifted to higher values with increasing nanotube content. Neat epoxy showed a Tg of approximately 78 ˚C, which rose to nearly 88˚C for the 0.75 wt% MWCNT composite. This increase is indicative of restricted segmental mobility in the polymer chains due to strong interfacial interactions with dispersed MWCNTs [19]-[24]. The drop in Tg at 1.0 wt% reinforces the hypothesis of poor dispersion and weaker interfacial adhesion at higher loadings.

Furthermore, the tan δ peak height decreased with MWCNT addition, pointing to increased material stiffness and energy dissipation capacity—features favorable for applications involving vibration damping [20] [23].

To assess the degree of interfacial bonding quantitatively, an interaction parameter (B) was calculated using the equation proposed by Ziegel and Romanov [21]. The highest B value corresponded to the 0.75 wt% sample, confirming optimal filler-matrix compatibility at this concentration.

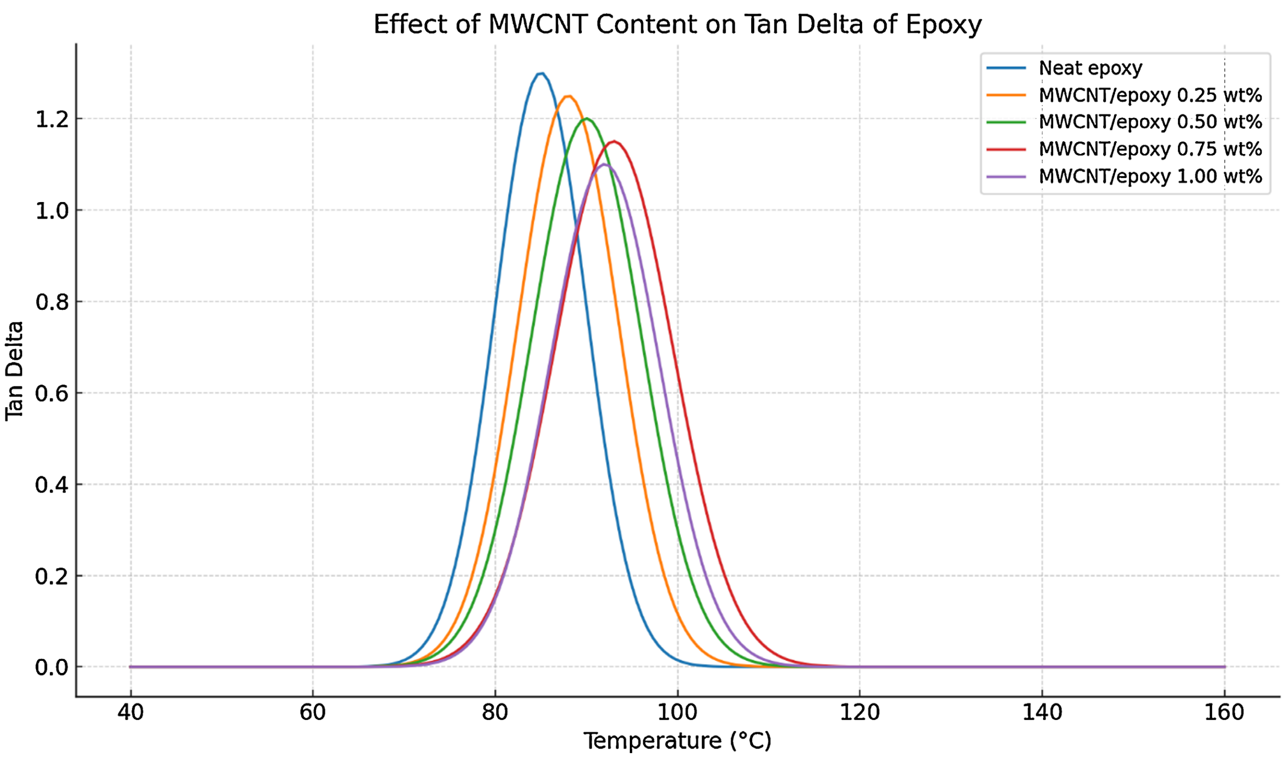

Figure 2. Variation of tan δ with temperature for different MWCNT loadings in epoxy matrix. The peak shift towards higher temperatures and the reduction in peak height signify increased glass transition temperature and material stiffness with MWCNT reinforcement.

3.2. Thermal Stability

The thermal stability of the composites was examined via thermogravimetric analysis. As illustrated in **Figure 3**, the onset of degradation, defined at 5% and 10% weight loss, shifted to higher temperatures as the MWCNT content increased. For example, at 0.75 wt% loading, the degradation temperature rose by nearly 15˚C compared to neat epoxy.

This improvement is ascribed to the barrier effect of MWCNTs, which inhibit the escape of volatile degradation products and delay thermal decomposition [25]. Moreover, the nanotubes may act as radical scavengers, suppressing the propagation of degradation reactions [24] [26]. The thermal behavior observed in this study aligns well with prior findings on carbon nanomaterial-reinforced polymers [27].

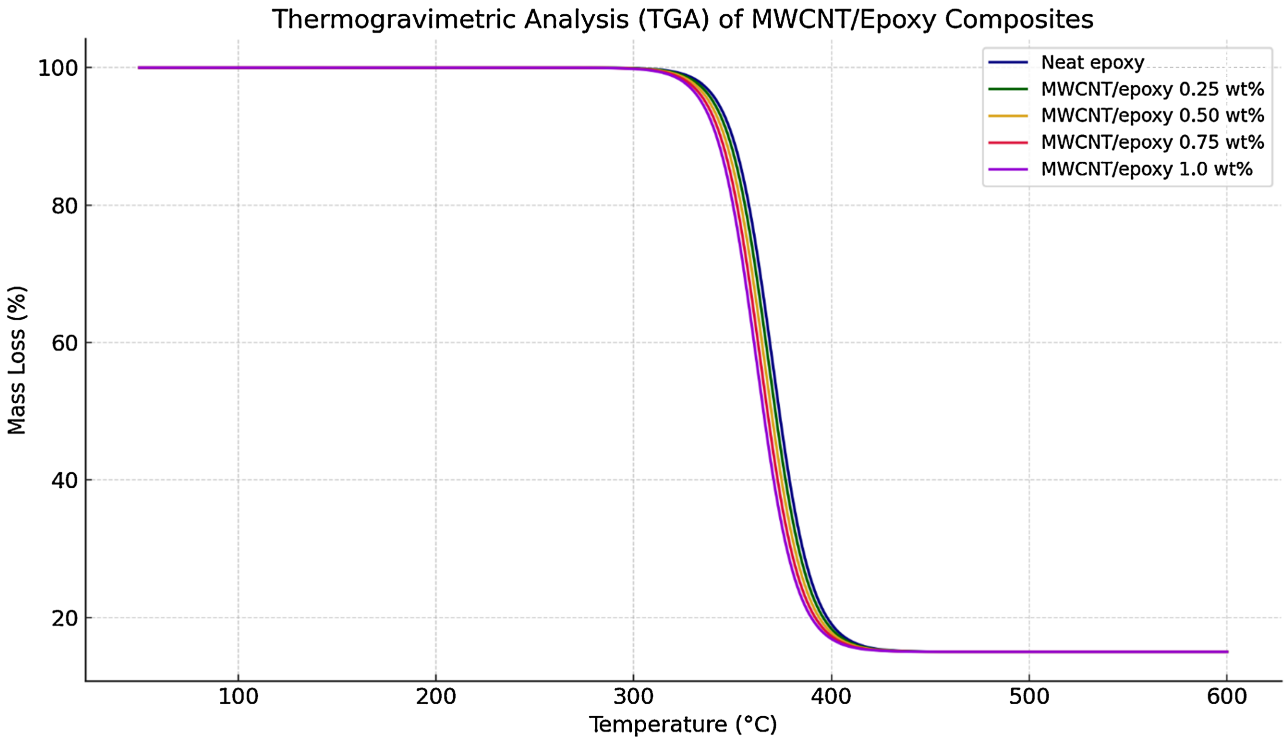

Figure 3. Thermogravimetric analysis (TGA) curves for neat epoxy and MWCNT/epoxy composites. The degradation temperatures increase with MWCNT addition up to 0.75 wt%, indicating enhanced thermal stability.

3.3. Fracture Surface Morphology

Field emission scanning electron microscopy (FESEM) images of the fracture surfaces (**Figure 4**) further confirmed the effect of dispersion on composite behavior. Neat epoxy exhibited smooth, river-like fracture patterns, characteristic of brittle failure. In contrast, the composites displayed rougher, more tortuous surfaces indicative of enhanced energy dissipation during fracture.

At 0.75 wt% MWCNTs, the fracture surface was densely populated with pulled-out nanotubes and deflected crack paths, implying strong matrix-filler interaction. At 1.0 wt%, however, visible clusters of MWCNTs were detected, acting as flaws that weaken the composite, consistent with the observed drop in mechanical performance.

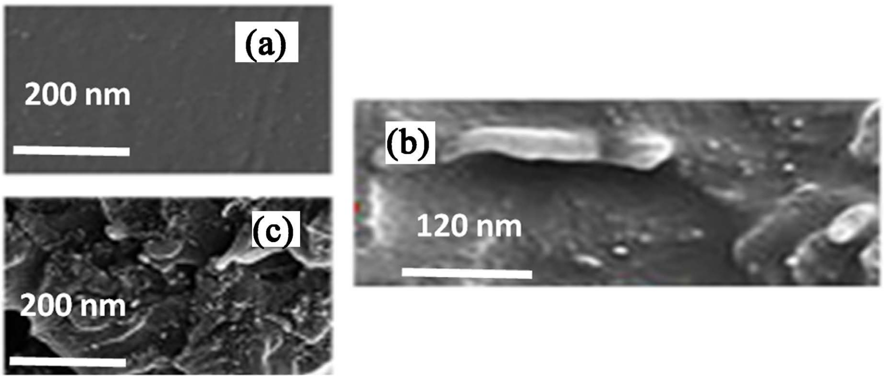

Figure 4. FESEM images of fracture surfaces: (a) neat epoxy, showing smooth and brittle fracture surface; (b) 0.75 wt%, displaying dense nanotube dispersion and rough fracture; (c) 1.00 wt%, where clustering of MWCNTs begins to appear, indicating reduced dispersion quality.

Such morphological features corroborate the mechanical and thermal trends and are in agreement with literature reporting that uniform nanotube dispersion contributes to increased fracture resistance [28] [29].

4. Conclusions

In this study, a dual-action dispersion technique employing ultrasonic waves and axial-flow shear mixing was successfully utilized to achieve homogeneous distribution of multi-walled carbon nanotubes (MWCNTs) within an epoxy matrix. The integration of this novel hybrid method demonstrated clear improvements in the thermo-mechanical performance of the resulting nanocomposites.

Dynamic mechanical analysis revealed that the storage modulus and glass transition temperature increased notably with MWCNT loading, peaking at 0.75 wt%, indicating enhanced stiffness and restricted molecular mobility due to strong interfacial bonding. Thermal stability, as assessed by TGA, also improved with increasing MWCNT content up to 0.75 wt%, owing to the barrier and radical-scavenging effects of well-dispersed nanotubes.

However, exceeding the 0.75 wt% threshold led to diminishing returns or slight reductions in all measured properties, attributed to the formation of MWCNT clusters that act as defects within the polymer matrix. FESEM observations of fracture surfaces corroborated these findings by visually confirming the transition from uniform dispersion to visible aggregation at higher nanotube loadings.

Overall, the findings suggest that the hybrid dispersion method is a highly effective, scalable approach for enhancing the mechanical and thermal characteristics of epoxy nanocomposites. The optimal MWCNT loading identified in this work (0.75 wt%) provides a benchmark for developing high-performance structural materials in aerospace, automotive, and advanced engineering applications.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

References

[1] Iijima, S. (1991) Helical Microtubules of Graphitic Carbon. *Nature*, **354**, 56-58. https://doi.org/10.1038/354056a0

[2] Baughman, R.H., Zakhidov, A.A. and de Heer, W.A. (2002) Carbon Nanotubes—The Route toward Applications. *Science*, **297**, 787-792. https://doi.org/10.1126/science.1060928

[3] Zheng, H., Zhang, W., Li, B., Zhu, J., Wang, C., Song, G., *et al.* (2022) Recent Advances of Interphases in Carbon Fiber-Reinforced Polymer Composites: A Review. *Composites Part B: Engineering*, **233**, Article ID: 109639. https://doi.org/10.1016/j.compositesb.2022.109639

[4] Spitalsky, Z., Tasis, D., Papagelis, K. and Galiotis, C. (2010) Carbon Nanotube-Polymer Composites: Chemistry, Processing, Mechanical and Electrical Properties. *Progress in Polymer Science*, **35**, 357-401. https://doi.org/10.1016/j.progpolymsci.2009.09.003

[5] Mohd Nurazzi, N., Asyraf, M.R.M., Khalina, A., Abdullah, N., Sabaruddin, F.A., Kamarudin, S.H., *et al.* (2021) Fabrication, Functionalization, and Application of Carbon Nanotube-Reinforced Polymer Composite: An Overview. *Polymers*, **13**, Article 1047. https://doi.org/10.3390/polym13071047

[6] Wang, Y., Tang, B., Gao, Y., Wu, X., Chen, J., Shan, L., *et al.* (2021) Epoxy Composites with High Thermal Conductivity by Constructing Three-Dimensional Carbon Fiber/Carbon/Nickel Networks Using an Electroplating Method. *ACS Omega*, **6**, 19238-19251. https://doi.org/10.1021/acsomega.1c02694

[7] Ogbonna, V.E., Popoola, A.P.I. and Popoola, O.M. (2022) A Review on Recent Advances on the Mechanical and Conductivity Properties of Epoxy Nanocomposites for Industrial Applications. *Polymer Bulletin*, **80**, 3449-3487. https://doi.org/10.1007/s00289-022-04249-4

[8] Peigney, A., Laurent, C., Flahaut, E., Bacsa, R.R. and Rousset, A. (2001) Specific Surface Area of Carbon Nanotubes and Bundles of Carbon Nanotubes. *Carbon*, **39**, 507-514. https://doi.org/10.1016/s0008-6223(00)00155-x

[9] Pegel, S., Pötschke, P., Petzold, G., Alig, I., Dudkin, S.M. and Lellinger, D. (2008) Dispersion, Agglomeration, and Network Formation of Multiwalled Carbon Nanotubes in Polycarbonate Melts. *Polymer*, **49**, 974-984. https://doi.org/10.1016/j.polymer.2007.12.024

[10] Kim, M.T., Rhee, K.Y., Park, S.J. and Hui, D. (2012) Effects of Silane-Modified Carbon Nanotubes on Flexural and Fracture Behaviors of Carbon Nanotube-Modified Epoxy/Basalt Composites. *Composites Part B: Engineering*, **43**, 2298-2302. https://doi.org/10.1016/j.compositesb.2011.12.007

[11] Abdalla, M., Dean, D., Adibempe, D., Nyairo, E., Robinson, P. and Thompson, G. (2007) The Effect of Interfacial Chemistry on Molecular Mobility and Morphology of Multiwalled Carbon Nanotubes Epoxy Nanocomposite. *Polymer*, **48**, 5662-5670. https://doi.org/10.1016/j.polymer.2007.06.073

[12] Albert, A.A., Parthasarathy, V. and Kumar, P.S. (2023) Review on Recent Progress in Epoxy-Based Composite Materials for electromagnetic Interference(EMI) Shielding Applications. *Polymer Composites*, **45**, 1956-1984. https://doi.org/10.1002/pc.27928

[13] Lachman, N. and Daniel Wagner, H. (2010) Correlation between Interfacial Molecular Structure and Mechanics in CNT/Epoxy Nano-Composites. *Composites Part A: Applied Science and Manufacturing*, **41**, 1093-1098. https://doi.org/10.1016/j.compositesa.2009.08.023

[14] Schulz, S.C., Faiella, G., Buschhorn, S.T., Prado, L.A.S.A., Giordano, M., Schulte, K., *et al.* (2011) Combined Electrical and Rheological Properties of Shear Induced Multiwall Carbon Nanotube Agglomerates in Epoxy Suspensions. *European Polymer Journal*, **47**, 2069-2077. https://doi.org/10.1016/j.eurpolymj.2011.07.022

[15] Thostenson, E.T. and Chou, T. (2002) Aligned Multi-Walled Carbon Nanotube-Reinforced Composites: Processing and Mechanical Characterization. *Journal of Physics D*: *Applied Physics*, **35**, L77-L80. https://doi.org/10.1088/0022-3727/35/16/103

[16] Guadagno, L., Naddeo, C., Vittoria, V., Sorrentino, A., Vertuccio, L., Raimondo, M., *et al.* (2010) Cure Behavior and Physical Properties of Epoxy Resin—Filled with Multiwalled Carbon Nanotubes. *Journal of Nanoscience and Nanotechnology*, **10**, 2686-2693. https://doi.org/10.1166/jnn.2010.1417

[17] Abdalla, M., Dean, D., Adibempe, D. and Nyairo, E. (2007) Interfacial Chemistry Effects on Morphology and Dynamics in MWCNT/Epoxy Nanocomposites. *Polymer*, **48**, 5662-5672.

[18] Guadagno, L., Vertuccio, L., Sorrentino, A., Raimondo, M., Naddeo, C., Vittoria, V., *et al.* (2009) Mechanical and Barrier Properties of Epoxy Resin Filled with Multi-Walled Carbon Nanotubes. *Carbon*, **47**, 2419-2430. https://doi.org/10.1016/j.carbon.2009.04.035

[19] Gojny, F.H. and Schulte, K. (2004) Functionalisation Effect on the Thermo-Mechanical Behaviour of Multi-Wall Carbon Nanotube/Epoxy-Composites. *Composites Science and Technology*, **64**, 2303-2308. https://doi.org/10.1016/j.compscitech.2004.01.024

[20] Jagtap, S., Rao, V.S. and Ratna, D. (2012) Preparation of Flexible Epoxy/Clay Nanocomposites: Effect of Preparation Method, Clay Modifier and Matrix Ductility. *Journal of Reinforced Plastics and Composites*, **32**, 183-196. https://doi.org/10.1177/0731684412462916

[21] Ziegel, K.D. and Romanov, A. (1973) Modulus Reinforcement in Elastomer Composites. I. Inorganic Fillers. *Journal of Applied Polymer Science*, **17**, 1119-1131. https://doi.org/10.1002/app.1973.070170410

[22] Rafiee, M.A., Rafiee, J., Wang, Z., Song, H., Yu, Z. and Koratkar, N. (2009) Enhanced Mechanical Properties of Nanocomposites at Low Graphene Content. *ACS Nano*, **3**, 3884-3890. https://doi.org/10.1021/nn9010472

[23] Curtin, W.A. and Sheldon, B.W. (2004) CNT-Reinforced Ceramics and Metals. *Materials Today*, **7**, 44-49. https://doi.org/10.1016/s1369-7021(04)00508-5

[24] Suarez, A.J., Urena, A. and Campo, M. (2013) Dispersion Techniques and Mechanical Behavior in MWCNT/Epoxy Composites. *Composites Part B*, **48**, 88-94.

[25] Harb, S.V., Pulcinelli, S.H. and Santilli, C.V. (2016) Reinforcement of Polymer Nanocomposites with Carbon Nanomaterials: Thermal Perspectives. *AACS Applied Materials & Interfaces Journal*, **8**, 16339-16350.

[26] Kumar, K., Ghosh, P.K. and Kumar, A. (2016) Enhancing Thermal and Mechanical Performance of TiO_2-Epoxy Nanocomposites. *Composites Part B*: *Engineering*, **97**, 353-360.

[27] Vertuccio, L., Guadagno, L., Spinelli, G., Lamberti, P. and Tucci, V. (2016) Nanotube-Modified Epoxy Composites for Structural Applications: Review of Thermal Effects. *Composites Part B*: *Engineering*, **107**, 192-202.

[28] Bortz, D.R., Heras, E.G. and Martin-Gullon, I. (2011) Impressive Fatigue Life and Fracture Toughness Improvements in Graphene Oxide/Epoxy Composites. *Macromolecules*, **45**, 238-245. https://doi.org/10.1021/ma201563k

[29] Kim, M.T., Rhee, K.Y., Lee, J.H., Hui, D. and Lau, A.K.T. (2011) Property Enhancement of a Carbon Fiber/Epoxy Composite by Using Carbon Nanotubes. *Composites Part B*: *Engineering*, **42**, 1257-1261.
https://doi.org/10.1016/j.compositesb.2011.02.005