

Effects of Halloysite Nano-Particles Reinforcement of Epoxy Composites Using a Diglycidyl Ether of Bisphenol-A Prepared by Ultrasonication Casting

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Abstract

This study explores a simple and operative approach for developing stronger epoxy-based composite materials by incorporating halloysite nanotubes (HNTs). The composites were made-up through hydraulic compression casting, combining ultrasonic mixing with applied pressure to produce consolidated composite sheets. HNTs were added to the epoxy resin at concentrations ranging from 1 to 5 wt.% at approximately 60°C, and the subsequent composites were examined to understand their internal structure, nanotube dispersion, and the presence of voids or defects. The findings establish that the incorporation of HNTs can significantly improve the physical and mechanical performance of the epoxy matrix. Among the compositions inspected, the composite containing 3 wt.% HNTs prepared with ultrasonic mixing exhibited the most favorable overall performance. Compared with neat epoxy, this preparation showed an approximately 9% increase in density and a substantial 34% enhancement in tensile strength. X-ray analysis further confirmed a comparatively uniform distribution of HNTs throughout the epoxy matrix, which contributed to the improved mechanical properties. In addition, good agreement between the experimental results and numerical simulations maintained the reliability of the findings. Overall, the study demonstrates that a moderately small addition of HNTs, particularly at 3 wt.%, can effectively enhance the density and tensile strength of epoxy composites. The planned fabrication method is straightforward and has potential for producing high-performance composite sheets for a range of engineering applications.

Keywords: Ultrasonication, epoxy, halloysite nanotubes, tensile analysis, finite component technique, XRD

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INTRODUCTION

Polymer-based composites, broadly categorized as thermosets and thermoplastics, are widely used because of their light weight combined with attractive mechanical performance. The final properties of thermoplastic composites are strongly governed by the processing conditions and the thermal response of their constituents during fabrication [1–5]. Thermoset polymers, in contrast, cure into a cross-linked three-dimensional network that generally resists deformation under load or elevated temperature [1]. Epoxy-based composites can be manufactured through a range of processing routes to achieve properties suited to a given application [1–5]. Reinforcements – like graphene nanoplatelets, clays, carbon based nano-tubes (CNT) and halloysite nanotubes (HNT) – are commonly used to enhance the resulting composite properties

[1–12]. Epoxy resin itself remains a preferred matrix material because of its favorable mechanical and physical properties combined with relatively low processing cost [3–6]. Halloysite nanotubes (HNT), a naturally occurring inorganic tubular clay, are particularly attractive as reinforcement for epoxy matrix composites for applications which demand a high ratio of strength-to-weight such as structural and automotive components [13–17]. There is growing interest in tailoring epoxy composite properties using naturally occurring tubular nanofillers such as CNT, SiO₂, and halloysite [16]. Such composites are typically processed by techniques including hand lay-up, powder compaction and ultrasonication-assisted casting [18, 19]. Previous studies [20–29] have demonstrated that the performance of epoxy/clay/HNT nanocomposites is strongly influenced by the interplay among filler content, dispersion quality, surface chemistry, curing behavior, and processing conditions. In particular, well-dispersed HNTs can enhance the mechanical strength and toughness of epoxy, while excessive filler loading or poor dispersion may reduce these benefits. Natural, low-cost reinforcements more broadly including agro-waste and lignocellulosic fillers have similarly been explored to improve the mechanical and moisture-resistance performance of polymer composites while reducing cost and environmental impact [30, 31].

Prior studies report that HNT addition improves properties that are mechanical in nature of epoxy matrices, with the improvement in impact-strength generally more pronounced than the corresponding improvements in tensile and flexural strength [12]. HNT is also lower in cost and non-toxic equated with last nanotubular fillers such as CNT [20]. It additionally shows good compatibility in food-packaging applications, where proper dispersion within the epoxy matrix has been linked to favorable chemical and physical performance [13–18].

Beyond HNT, other nano- and micro-scale reinforcements – such as natural fiber fillers [32], nano-fiber-reinforced polyester systems [33], and clay/HNT-type fillers – have been used to enhance the mechanical, morphological and dynamic-mechanical performance of epoxy and polyester matrices, with the choice of filler generally reflecting a trade-off between reinforcement efficiency, dispersion difficulty and cost. Real-time monitoring approaches are increasingly proposed to track the in-service performance and durability of polymer composites of this type, offering a complementary route to validate the long-term structural predictions made here [34]. Filler incorporation has been reported to reduce water absorption in related fiber/filler-reinforced polyester systems as packing density increases and porosity falls [31], while fiber type, orientation and stacking sequence have been shown to govern moisture uptake in hybrid fiber–metal epoxy laminates [35]. The dispersion quality of HNT strongly affects the ultimate properties and processability of nano-composites, a systematic study considering HNT-reinforced epoxy is needed to clarify key processing questions, particularly the optimal mixing procedure and the choice of curing agent. To achieve uniform dispersion of HNT within the epoxy matrix, ultrasonication mixing was employed while preparing HNT/x (x = 0–5 wt.%) reinforced composites, using “triethylenetetramine” (TETA) as the curing representative, and the consequences on epoxy matrix belongings were estimated. Impact properties of the composites were compared across the different HNT loadings, and microstructural examination of the fabricated specimens confirmed the effectiveness of the HNT/epoxy system, showing a progressive increase in density when the concentration was raised from 1 to 5 wt.% of reinforced epoxy HNT.

Conventional composites typically need high filler loadings (20–60 wt.%) of micron-scale reinforcement to achieve meaningful property gains, which adds weight, increases brittleness, and can degrade processability. Polymer nanocomposites achieve comparable or superior improvements at much lower loadings (typically 1–5 wt.%, as used in this study) because:

- *Enormous Interfacial Area Per Unit Mass:* Nanoscale fillers have vastly higher surface-area-to-volume ratios, so even a small mass fraction provides a large filler–matrix contact area for stress transfer.
- *Multifunctional Gains:* Simultaneous improvements in mechanical strength/toughness, thermal stability, and barrier resistance, without the density penalty of heavy conventional loading.
- *Preserved Ductility and Processability:* Low volume fractions keep viscosity and matrix mobility closer to the neat resin, easing fabrication (relevant to the ultrasonication/casting route used here).

- *Superior Crack-Resistance Mechanisms:* Nanoparticles promote crack deflection, pinning, and micro-shear yielding at the crack tip, mechanisms that are far less effective at the micron scale (consistent with your SEM observations in Section 6).

MATERIALS AND PROCEDURES

Collection of Compound Materials

The Diglycidyl ether of bisphenol-A epoxy (DGEBA, LY 556) was used as the matrix material, triethylenetetramine (TETA, HY 951, Ciba-Geigy Ltd., Mumbai, India) served as the curing agent/hardener, and halloysite nanotube particulate filler (Sigma-Aldrich, USA) was used as reinforcement to prepare samples with varying HNT loading. The halloysite filler and the epoxy matrix properties are summarized in Tables 1 and 2, respectively.

Table 1. Properties of the halloysite nanotube filler.

Halloysite nanotubular in nature (HNT's)	
Type	Kaolinite
Chemical Formula	Halloysite- $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4 \cdot n\text{H}_2\text{O}$.
Symmetry of the crystal	Mono-clinic.
Sp.gr	2.01–2.67 (2.54 in Existing Work).

Table 2. Selected properties of the epoxy resin and their origin.

Features	Aim
High reactivity	Attributed to the hydroxyl groups present in the resin.
Chemical stability	Result of the ether linkages in the epoxy backbone.
Excellent adhesion to surfaces	Arises from strong interfacial attraction between the resin and the substrate surface.
Outstanding electrical resistivity	Due to its great di-electric power.

HNT/Epoxy composite Planning

The fabrication route strongly influences the final properties of the halloysite-reinforced epoxy nanocomposite. Samples of 150 mm × 150 mm × 3 mm were prepared for each HNT/x (x = 1–5 wt.%) formulation, with the resin-filler mixtures subjected to ultrasonication (Rivotek sonicator, 700 W, model XT 546, 50% amplitude) for 1 hour to achieve a uniform morphology (Table 3). The HNT-epoxy blend was then discharged into a steel mould and at a room temperature amid 30–35°C was cured under a hydraulic press at 1600 PSI for 24 hours. Specimens were cut to shape using a contour-cutting device in accordance with ASTM standards for the dissimilar mechanical tests (Figure 1).

Table 3. Sample codes and formulation of the composites.

Code of sample	Reinforcement	Resin of epoxy (grams)	Preserving material (grams)
Sample 00	Preserved unchanged	150	15
Sample 01	Preserved 1 weight.% HNT	150	15
Sample 02	Preserved d 2 weight.% HNT	150	15
Sample 03	Preserved 3 weight.% HNT	150	15
Sample 04	Preserved 4 weight.% HNT	150	15
Sample 05	Preserved 5 weight.% HNT	150	15



Figure 1. Photograph of the fabricated HNT/epoxy composite specimens.

MECHANICAL EXAMINATION OF SPECIMEN

Impact Examination

The belongings impact in nature remained measured on an Izod impact testing machine (Tinius Olsen) with a 2.7 J capacity hammer at the temperature of room environment, following the ASTM D256 method-A norms, with striker impacting the HNT/epoxy specimen at approximately 3.5 m/s. Five specimens were tested for each HNT/x ($x = 0\text{--}5\text{ wt.}\%$) composite to ensure reliable results.

Modeling and Finite Component Examination Method

Numerical modelling is increasingly used to describe composite material behavior, although software predictions must still be validated against experimental data. The present study outlines analysis based on finite elements that were implemented to model the structural response and strength of the fabricated composite, which was assumed to behave as a fully isotropic material.

Modeling Based on Impact Examination

The modeling based on FEM analysis for the impact examination, a CAD model of each composite specimen was built in Autodesk Inventor 2016 and exported in IGES format. The IGES file was then imported into Ansys, meshed using the Ansys Mesh element, and analyzed by the Ansys Autodyne component on the Ansys Workbench platform. An explicit dynamics approach was adopted to model the elastic response of the specimen under impact, since this method is well suited to the linear and nonlinear structural mechanics involved in high-speed impact problems.

Explicit dynamics is well suited to simulating events that occur over very short time scales. The mesh generated for the impact model consisted of 914 nodules and 3575 quadrangular and trilateral components (Figure 2).

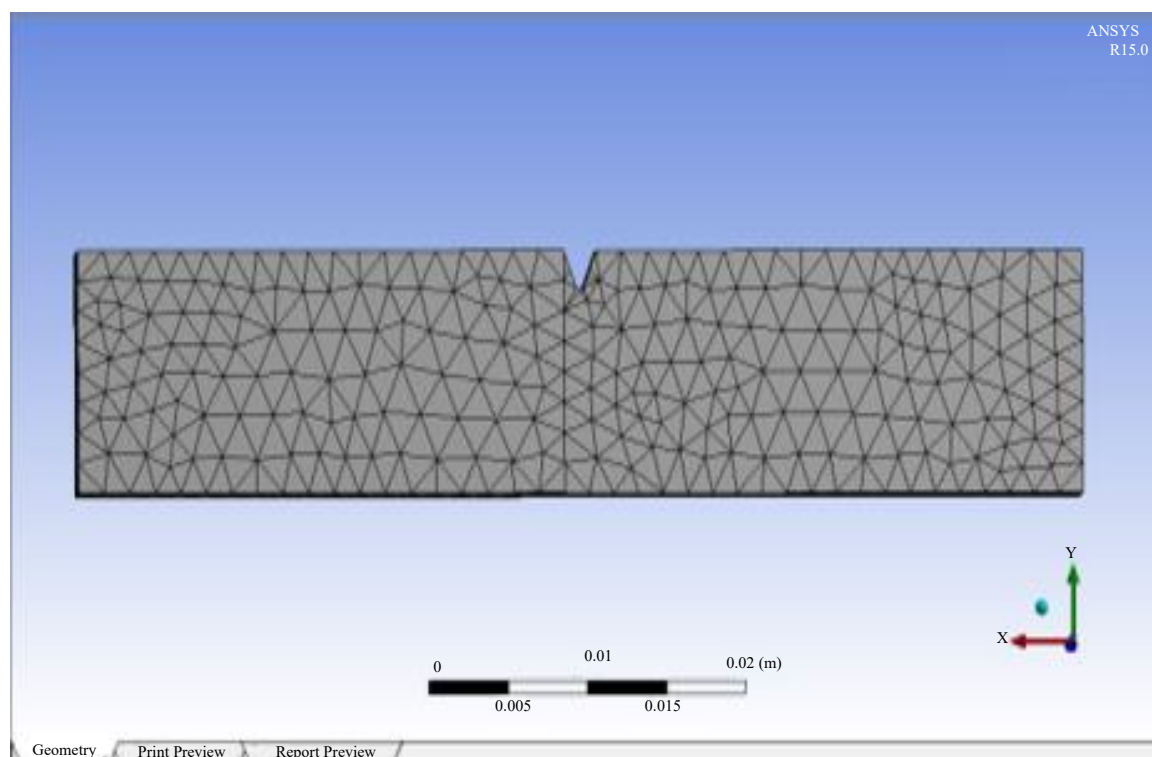


Figure 2. Finite element mesh generated for the Izod impact test model.

INVESTIGATIONAL OUTCOMES AND CONVERSATION

Figure 3 presents the power of impact on the epoxy Halloysite nanotubes with variable (x), i.e., percentage of HNT in epoxy varying from $x = 0\%$ wt. to $x = 5\%$ wt.

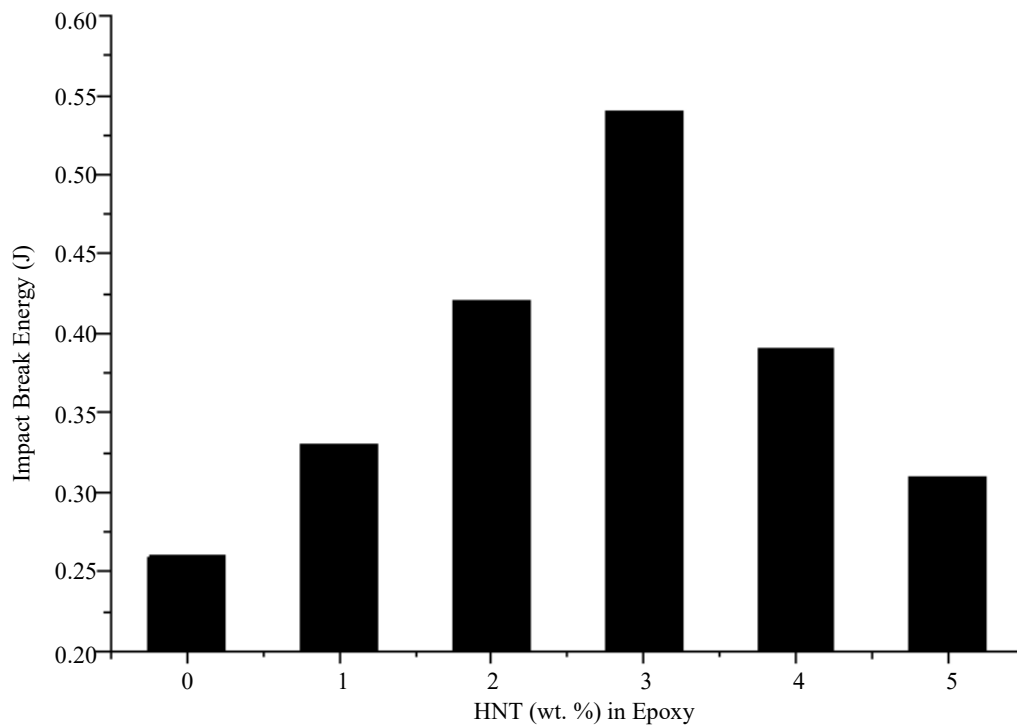


Figure 3. Change in disruption energy during impact with halloysite filling.

The results indicate that incorporating 3 wt.% HNT into the epoxy matrix increased the impact disruption energy by almost 3.5 times, from 0.33 J for the neat epoxy to 1.189 J, as shown in Figure 4.

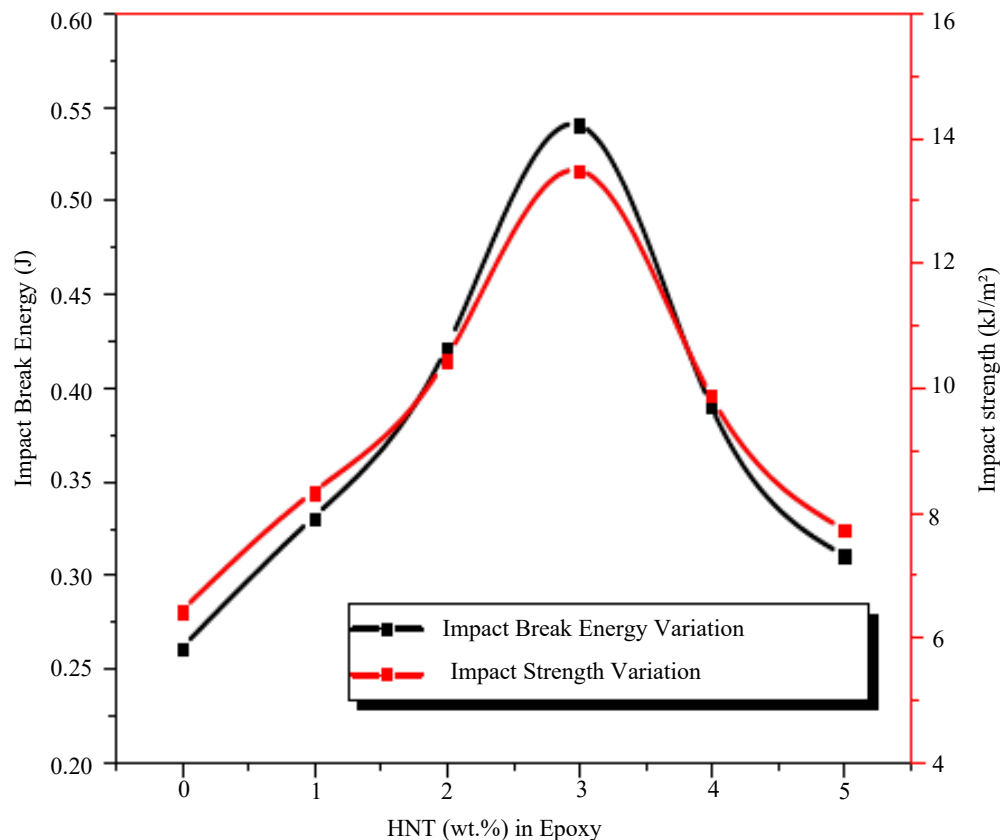


Figure 4. Comparison of power of impact and impact energy disruption across halloysite loadings.

This improvement in impact strength is accredited to the larger contact area of the interface amid HNT and epoxy matrix, which allows the composite to engage additional energy throughout impact filling (Table 4).

Table 4. Results of strength of impact for the fabricated composites.

Code of sample	Strength of impact (kJ/m ²)		
	Test range		Deviation from the mean value
	Min value	Max value	
Sample 00	5.68	7.20	6.4 ± 0.8
Sample 01	7.57	9.63	8.31 ± 1.3
Sample 02	9.50	11.46	10.43 ± 1
Sample 03	13.04	13.87	13.47 ± 0.5
Sample 04	9.22	10.45	9.86 ± 0.5
Sample 05	7.55	7.95	7.72 ± 0.3

The unvarying dispersal of HNT within the matrix of the polymer is accountable for the upper energy of impact that was engaged for failure previously [36]. It is worth noting that the observed increase now energy absorbed through impact failure is analogous to the hardening outcome typically achieved with rubber modifiers [37], yet a similar level of toughening was reached here at a much lower reinforcement content of just 3 wt.% HNT [6, 38].

FINITE COMPONENT MODEL OUTCOMES AND DISCUSSION

The disproportionate effect of low nanofiller loadings comes from several compounding mechanisms:

- High aspect ratio and surface area mean even a small mass fraction generates a large interfacial area for load transfer.
- Inter-particle spacing at the nanoscale is small even at low volume fraction, so the filler can approach a network-like distribution that resists local deformation.
- An “interphase” region of restricted polymer chain mobility forms around each nanoparticle, locally stiffening the matrix.
- Energy-dissipating mechanisms crack deflection/bridging by the tubular HNT particles and micro-cracking/shear yielding at the interface, Figure 8 absorbs and redistributes energy impact.
- This also explains your own trend: below 3 wt.% there is not enough reinforcement network; above 3 wt.% partagglomerate, creating voids and stress concentrators that reverse the gain which is exactly the optimum you report.

Figure 5 demonstrates energy absorbed through impact failure of the HNT/epoxy nano-composites. The complete set of simulations for the composite geometries was executed by the non-linear finite module solver LS-DYNA. The Impact examination outcomes found from the finite component examine in Ansys-15.0 for the HNT or epoxy compound samples are conferred below [39, 40].

For the Sample-03 composite model, simulation results show that the impactor carries 2.7 Joule of energy kinetic in nature previously impinging the sample with 2.23 Joule subsequently impact, the reduction reflecting the energy absorbed and dissipated as the specimen fractures. The impact energy required to break the composite increases steadily as HNT content is raised in the epoxy/Halloysite Nano-Tubes/x mixtures (x = 0 to 5%weight,) which is attributed to the greater amount of reinforcing HNT particle available to absorb impact energy across the epoxy matrix [41]. Table 5 compares the investigational and finite component (Ansys Workbench unit 15.0) outcomes for the impact examination of each compound. For the 3 wt.% HNT composites, the experimentally measured impact fracture energy is 0.54 J, compared with an Autodyne solver estimate of 0.4658 J from the Ansys Workbench simulation.

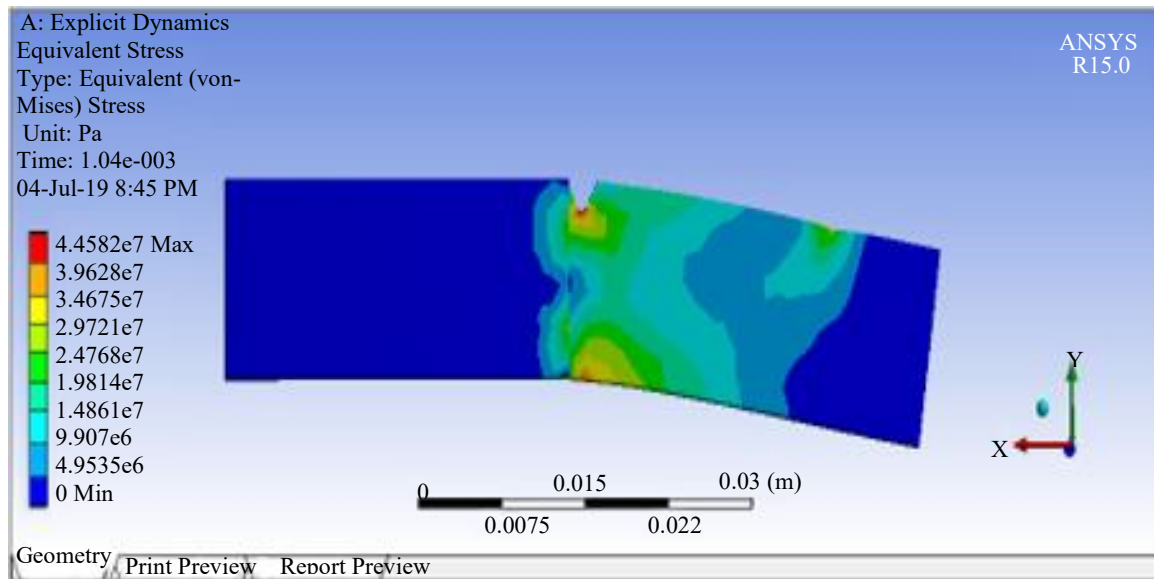


Figure 5. Simulated stress distribution and failure of the composite in the Izod impact test.

Table 5. Comparison of investigational and finite component impact examination outcomes.

Sample code	Impact breakdown energy (joule) obtained through experiments	Impact breakdown energy (joule) (through simulation on ANSYS)	Error (%)
Sample 00	0.29	$2.7013 - 2.4332 = 0.2681$	3.11
Sample 01	0.34	$2.7013 - 2.3849 = 0.3164$	4.12
Sample 02	0.44	$2.7013 - 2.3237 = 0.3776$	10.09
Sample 03	0.56	$2.7013 - 2.2355 = 0.4658$	13.74
Sample 04	0.40	$2.7013 - 2.3008 = 0.4005$	2.69
Sample 05	0.34	$2.7013 - 2.3701 = 0.3312$	6.84

For the unmodified epoxy, the experimentally measured impact energy is 0.26 J, compared with a predicted Autodyne solver value of 0.2681 J, as displayed with the help of Figure 6.

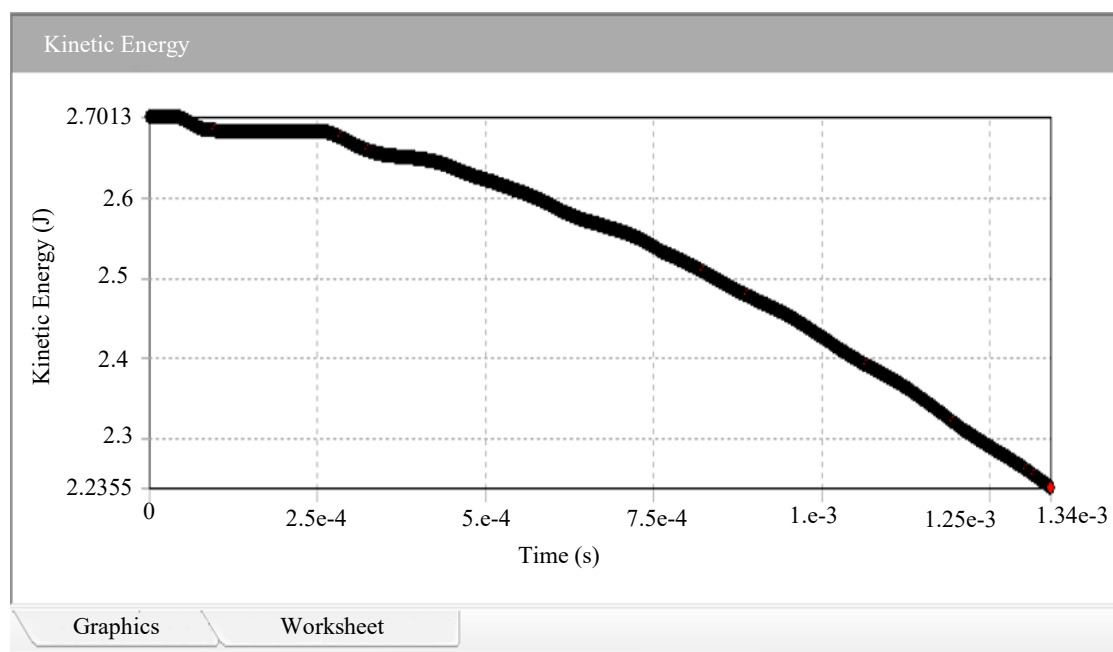


Figure 6. Simulated impact breakdown energy history for the Sample-03 compound.

Overall, the finite element impact test results follow the same trend as the experimental data. The discrepancies between the two can be accredited to fabrication defects like matrix non-uniformity, voids spaces, and weak bonding between the HNT-epoxy resin particles. The largest deviation between experimental and numerical impact break energy values was 13.74%. HNT offers a favorable balance of low cost, natural abundance, non-toxicity and ease of dispersion without chemical modification, at the expense of the very largest mechanical/electrical gains achievable (at greater cost and processing difficulty) with graphene or CNT. This trade-off is the basis for selecting HNT in the present study. Halloysite is a hydrated aluminosilicate clay ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4 \cdot n\text{H}_2\text{O}$), so it carries structural hydroxyl groups that are intrinsically hydrophilic. Its net effect on composite water uptake is dispersion-dependent, not fixed in one direction:

- At low loading with good dispersion (as achieved here via ultrasonication up to 3 wt.%), the tubular HNT particles increase the tortuosity of the moisture diffusion path through the epoxy, which can reduce the rate of water uptake relative to the neat resin.
- At higher loading or where agglomeration occurs (as your own results show above 3 wt.%), HNT clusters and the associated interfacial micro-voids act as capillary pathways and moisture-absorption sites, increasing water uptake and accelerating hydrolytic degradation of the filler–matrix interface over time.

SCANNING ELECTRON MICROSCOPY OBSERVATION

The Scanning Electron Microscopy images of the fractured tensile specimen were examined to assess failure behavior of the HNT/epoxy composites. The unmodified epoxy shows a flaky fracture morphology (Figure 7) showing dispersed HNT clusters

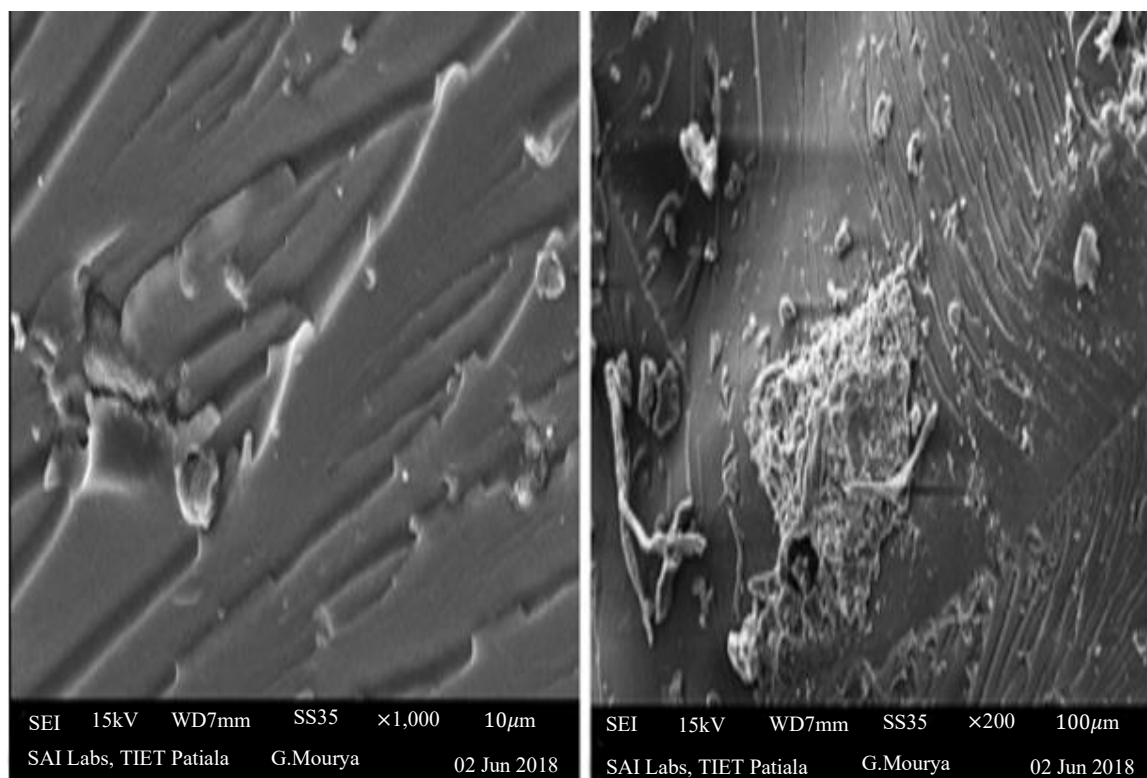


Figure 7. Fracture surface micrographs of HNT/epoxy composites: (a) 0 wt.% HNT, (b) 3 wt.% HNT.

The SEM micrographs (a) and (b) show that the 3 wt.% HNT sample achieves excellent diffusion of the nano-tubes throughout the matrix of polymer, with individual halloysite nano-tubes clearly visible. SEM imaging was used to study how these nanotubes influence crack commencement and propagation in the HNT/epoxy nanocomposite. The fracture surface of the modified composite shows evidence of

extensive micro-cracking and shear yielding, mechanisms that dissipate breakage energy and resist crack development, forming a stronger fracture network than the unmodified epoxy. This behavior, together with the favorable properties of HNT throughout the matrix, accounts for the property improvements observed at 3 wt.% loading (Figure 8). Above 3 wt.%, HNT tends to agglomerate, which reduces strength.

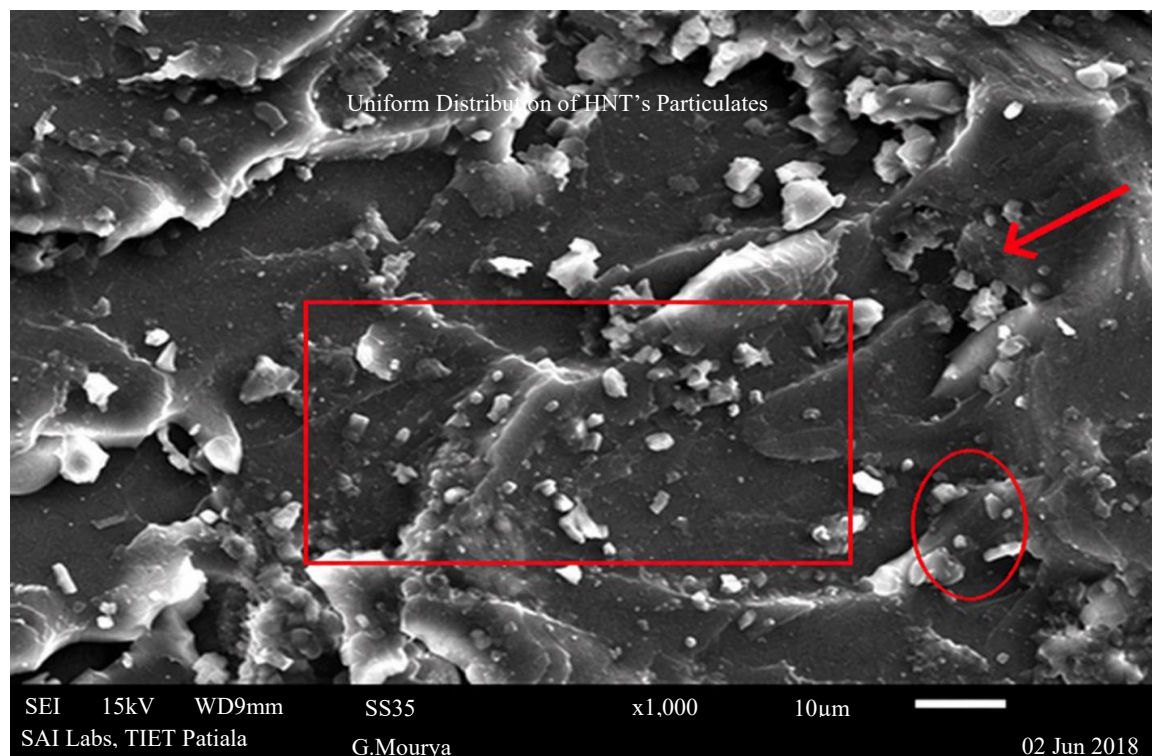


Figure 8. Micrograph of the nanocomposite at 3 wt.% HNT loading.

The key experimental finding is that 3 wt.% HNT gives the best impact properties compared with the neighboring HNT loadings. The lower performance at other loadings is likely due to the existence of little cavities and irregular circulation of halloysite nanotubes inside the epoxy matrix.

CONCLUSIONS

- Impact strength enhanced significantly with HNT reinforcement, with the 3 wt.% HNT/epoxy nanocomposite showing a 109.87% increase compared with unmodified epoxy.
- 3 wt.% HNT was the optimum concentration, as impact break energy amplified up to this level and then decreased at 4 wt.% and 5 wt.% HNT.
- The enhanced impact performance was mainly due to the toughening effect of HNT particles, which helped absorb impact energy and restrict crack propagation.
- The SEM analysis confirmed improved damage resistance in the HNT-reinforced composite, with reduced cracking and particle damage and greater shear-band development compared with unmodified epoxy.
- ANSYS finite element simulations using the Design and Autodyn modules showed good agreement with the experimental results and successfully predicted the overall impact behavior of the HNT/epoxy nanocomposites.

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