

Effect of Nanoclay Loading on Physical and Mechanical Properties of Epoxy Composites

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Abstract

The effects of multilayer silicate loading on hardness, tensile, and impact properties were examined by developing epoxy–nanoclay composites with 0–5 wt% nanoclay. A desirable compromise between reduced void content and higher Shore D hardness was reached with an ideal nanoclay loading of 3 wt%, which reflected improved load transfer and increased matrix stiffness as a result of limited polymer chain mobility. Tensile strength and modulus rose up to 3 wt% nanoclay, aligning with micromechanical predictions from the Halpin–Tsai model for high aspect-ratio platelet reinforcements. Impact strength exhibited a 56% enhancement at 3 wt% due to synergistic fracture toughening mechanisms, including crack pinning, deflection, branching, and localized plastic shear, whereas a reduction at 5 wt% was ascribed to platelet tactoid aggregation and void-induced stress concentrations. The statistical study demonstrated that the 3 wt% composition shown markedly enhanced impact resistance. Overall, the findings show that mechanical reinforcement is maximized at an ideal nanoclay loading of 3 wt% before aggregation-driven embrittlement imposes declining returns.

Keywords: Epoxy-nanoclay composites, hardness, tensile properties, impact strength

INTRODUCTION

Owing to the increasing demand for lightweight materials with enhanced mechanical performance, polymer matrix composites have been thoroughly investigated for engineering applications such as automotive, aerospace, and marine structures [1]. Epoxy resins have attracted a lot of research interest due to its exceptional mechanical properties, chemical stability, and variety of uses. However, pristine epoxy cannot be utilized directly in high-load applications due to its poor strength and hardness. Enhancing epoxy's mechanical performance using nanoscale fillers has been a key area of research since these materials can greatly increase stiffness, strength, toughness, and other mechanical properties through increased interfacial contact and load transfer efficiency [2, 3].

The use of nanoscale fillers is a successful method for improving the mechanical and functional properties of polymer matrices [4]. One of the most popular nanofillers is nanoclay particles due to its high aspect ratio, low cost, and ability to increase stiffness, hardness, and impact resistance. When dispersed uniformly, nanoclay can significantly change the microstructure and mechanical behavior of epoxy systems.

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The effects of various nanofillers, such as silica, graphene, carbon nanotubes, nanoclay, and titanium dioxide, on the mechanical characteristics of epoxy composites, with an emphasis on hardness, tensile strength, elastic modulus, and impact resistance [5–10]. Nano-silica additions can improve tensile strength, modulus, and fracture toughness due to their huge surface area and efficient stress transmission [5]. Functionalization of silica nanoparticles enhances the mechanical performance of epoxy nanocomposites in comparison to

untreated nanoparticles [6]. MWCNT reinforcement can improve modulus and tensile strength if adequate dispersion is achieved [7]. The alignment of CNTs significantly enhances the mechanical characteristics of epoxy nanocomposites when compared to randomly oriented CNT systems, leading to increased quasi-static fracture toughness and elastic modulus. These developments are primarily due to efficient load transmission, crack-tip deflection, and CNT pull-out mechanisms that perform well even at very low CNT concentrations. [8]. Graphene and graphene oxide (GO) fillers greatly improve tensile and flexural properties due to their high intrinsic strength and excellent interfacial adhesion [9]. Greater graphene loading typically results in greater mechanical stiffness; however, if this loading beyond a critical threshold, agglomeration issues may arise [1, 10]. Hybrid systems of carbon nanotubes (CNTs) and graphene nanoplatelets (GNPs) show synergistic advantages in mechanical behavior when compared to single filler systems [11]. Nanoclay platelets improve stiffness and strength due to their high aspect ratio and barrier qualities against fracture propagation [12]. Epoxy-nanoclay systems' mechanical characteristics are determined by their dispersion quality and clay concentration; excessive clay can result in agglomeration, which reduces strength [1, 13]. Two-dimensional nanomaterials (such as graphene and boron nitride) often outperform zero-dimensional nanofillers in mechanical enhancements [14].

Despite these benefits, filler content, dispersion quality, and interfacial bonding have a significant impact on epoxy–nanoclay composite performance [12, 13]. Premature failure may result from poor nanoclay particle dispersion and aggregation, which can operate as stress concentrators. Although many studies have looked at epoxy composites reinforced with various nanofillers, such as graphene, carbon nanotubes, nano-silica, and nanoclay, a comprehensive understanding of the unique impact of nanoclay loading on the mechanical and physical properties of epoxy composites is still lacking. The bulk of current research focuses on hybrid nanofiller systems, surface-modified fillers, or sophisticated dispersion techniques, however systematic studies employing simple manufacturing processes with controlled fluctuations in nanoclay content are quite uncommon. Inconsistent trends in tensile and impact properties described in the literature further emphasize the need for more experimental clarification on the effects of nanoclay dispersion and agglomeration at different filler loadings.

This work is the first thorough investigation of epoxy composites augmented just with nanoclay using a conventional casting method throughout a predetermined range of filler concentrations. In order to offer a clear and thorough understanding of the evolution of both surface and bulk properties as a function of nanoclay concentration, this work focuses on the simultaneous evaluation of density, characteristic hardness, tensile strength, and impact strength. Therefore, it is crucial to conduct a thorough examination into how the amount of nanoclay affects mechanical properties. The goal of the current study is to assess how different nanoclay contents affect the impact, tensile, and hardness characteristics of epoxy composites made by a straightforward casting method.

Because it identifies the optimal nanoclay loading range that enhances surface hardness, strength, and impact resistance while highlighting the limitations imposed by agglomeration on bulk mechanical performance, this research is important and helpful. The findings provide helpful suggestions for the development of low-cost, lightweight epoxy nanocomposites suitable for structural and semi-structural applications where improved surface properties and material efficiency are required without requiring complex processing methods.

METHODOLOGY

Materials

Lapox L-12 epoxy resin, a thermosetting polymer based on Diglycidyl ether of Bisphenol-A (DGEBA), served as the matrix material for this study. The resin was selected due to its extensive use in structural and technical applications, high mechanical strength, excellent adhesive qualities, and resistance to chemicals. It was acquired from Pune, India's Atul Ltd. The same manufacturer's K-6 hardener was used to initiate the epoxy resin's cross-linking reaction. The epoxy resin and hardener

were mixed in accordance with the manufacturer's recommended ratio to ensure complete curing and consistent, repeatable material properties.

Fabrication

Epoxy-nanoclay composites were developed using a liquid-phase mixing process to ensure the integration of nanoclay particles (1 to 5 wt%) into the polymer matrix. The entire method of creating epoxy-based nanocomposites is shown in Figure 1, and it is described in detail below.

The amount of epoxy resin that was required was weighed using an electronic balance. Nanoclay particles were gradually incorporated into the resin. To achieve an initial level of dispersion and prevent the formation of large clusters, the mixture was manually stirred for 15 to 20 min.

To ensure a completely homogeneous nanocomposite and enhance the exfoliation of the nanoclay layers, the mixture was ultrasonically sonicated. Inside the epoxy, disintegrating nanoclay agglomerates into distinct platelets or intercalated formations when cavitation bubbles produced by high-frequency ultrasonic vibrations rupture and release energy.

Following the dispersion step, K-6 hardener was applied to the modified resin. The chemicals were well mixed in order to initiate the cross-linking process. In order to prevent trapped air bubbles from serving as stress concentrators in the final composite, care was taken during this phase. A Teflon mould measuring $150 \times 100 \times 3 \text{ mm}^3$ was filled with the completed resin mix. In a controlled environment, the curing cycle was run for 6 h at 80°C . This high temperature ensures that the chemical reaction between the epoxy and the hardener is completed.

After the curing phase, the mould was allowed to naturally cool to room temperature in order to avoid internal thermal stresses. The solid composite plates were removed from the mould and then precisely cut using tools to meet the requirements for tensile and impact testing. The composition of all epoxy nanocomposites made by adding various nanoclay loadings to the epoxy matrix is compiled in Table 1.

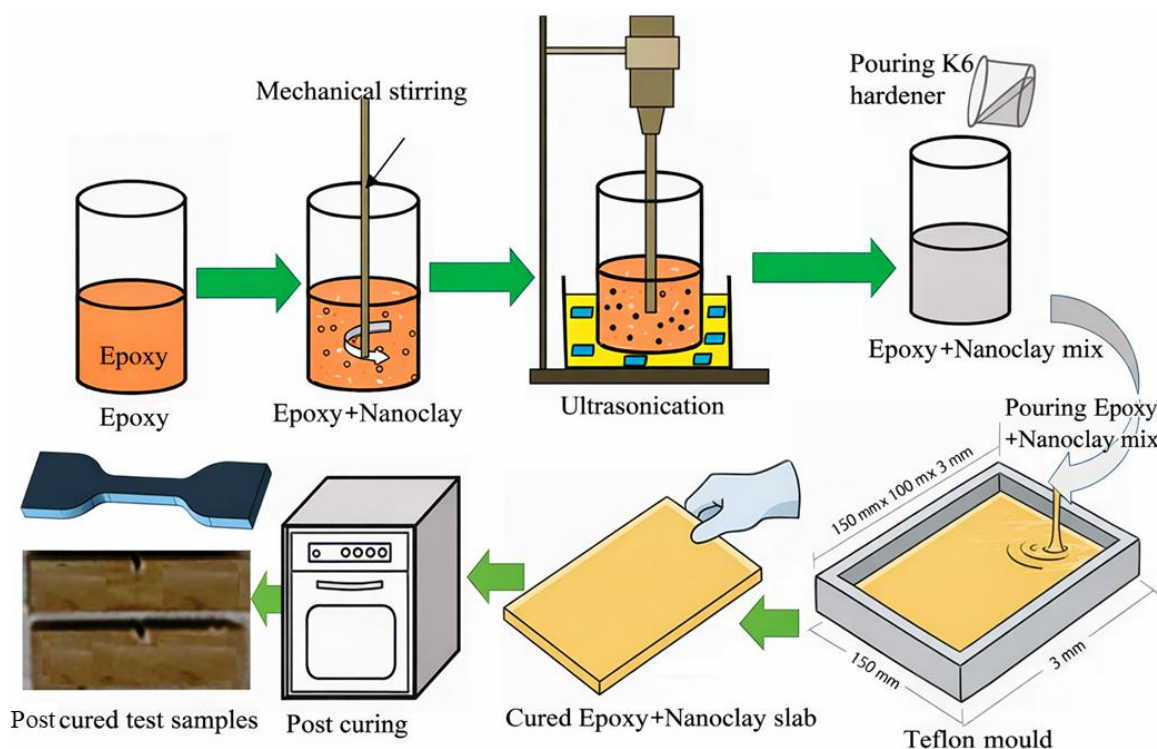


Figure 1. Schematic representation of the fabrication process for epoxy nanocomposites.

Table 1. The composition of all epoxy nanocomposites produced in the present work.

| S. No. | Composites | Nanoclay* (wt%) |
|--------|-----------------------|-----------------|
| 1 | Epoxy* | --- |
| 2 | Epoxy +1 wt% Nanoclay | 1 |
| 3 | Epoxy +3 wt% Nanoclay | 3 |
| 4 | Epoxy +5 wt% Nanoclay | 5 |

*Supplier's density data: Epoxy 1.16 g/cm³, Nanoclay 1.77 g/cm³

Physico-Mechanical Testing

Density Measurement

Pristine epoxy and epoxy–nanoclay composite samples were assessed for density using a densitometer based on Archimedes' principle in accordance with ASTM D792 [15]. Each specimen was weighed in the air before being completely immersed in room temperature distilled water. To ensure precision and consistency, three measurements were taken for each mixture. After that, the average outcome was presented. Equation (1) was used to compute the composite's density.

$$\rho_c = \frac{w_a}{(w_a - w_w)} \times \rho_w \quad (1)$$

where

ρ_c and ρ_{cw} are density of the composite and water respectively; w_a and w_w are the weight of the sample in air and water respectively.

The volume fraction of voids in percentage (V_v) of the composite was given by equation (2).

$$V_v = \frac{(\rho_t - \rho_c)}{\rho_t} \times 100 \quad (2)$$

where

ρ_t and ρ_c are theoretical and measured density of the composite respectively.

Hardness Testing

Hardness measurements were performed in compliance with ASTM D2240 [16] using a Shore hardness tester. To lessen localized effects, ten indentations were made at different locations on the specimen surface for each set of samples. Each indentation was created with a dwell time of 15 seconds and a 50 gf load. The average shore hardness value and standard deviation were given.

Tensile Testing

For tensile testing, a Kalpak Universal Testing Machine (UTM) was used in accordance with ASTM D638 [17]. Pristine epoxy and three sets of specimens reflecting different nanoclay loadings were created. To ensure statistical reliability and repeatability, three specimens were evaluated from each set. A constant crosshead speed of 3 mm/min was used for all tensile tests, which were carried out at room temperature. The tensile strength and modulus findings are the average of three measurements; the accompanying standard deviation was calculated to assess data dispersion.

Izod Impact Testing

Impact strength was evaluated using the Izod impact test in accordance with ASTM D256 [18]. Standard notched specimens were created using a notch cutter in compliance with the standard. The impact strength values shown are the average of the tested samples, with the standard deviation adjusted to account for experimental variability. Two specimens were analyzed for each composition.

RESULTS AND DISCUSSION

Density and Voids Analysis of Nanoclay Modified Epoxy Composites

The theoretical and experimental densities of the produced epoxy - nanoclay composites were compared in order to assess their physical characteristics.

Table 2. Measured density and voids of epoxy-nanoclay composites.

| Composites | Theoretical density (g/cm ³) | Measured density (g/cm ³) | Voids (%) |
|-----------------------|--|---------------------------------------|-----------|
| Epoxy | 1.160 | 1.156 | 0.345 |
| Epoxy +1 wt% Nanoclay | 1.164 | 1.158 | 0.515 |
| Epoxy +3 wt% Nanoclay | 1.172 | 1.165 | 0.597 |
| Epoxy +5 wt% Nanoclay | 1.180 | 1.168 | 1.017 |

The Archimedes principle was used to determine the experimental density, whilst the rule of mixtures was used to get the theoretical density. Due to the increased density of the silicate nanolayers (1.77 g/cm³) compared to the epoxy matrix (1.16 g/cm³), a progressive increase in density was seen with increasing nanoclay loading, as shown in Table 2.

The difference between theoretical and measured values was used to calculate the void content, a crucial statistic for evaluating the structural integrity of composites. The baseline void content of 0.345% in the virgin epoxy demonstrated a superior casting process with less air entrapment. The void content stayed mostly constant at 0.515% and 0.597%, respectively, after adding 1 wt% and 3 wt% nanoclay. At the 5 wt% loading level, however, a notable increase to 1.017% was noted.

The reinforced composites show a definite trade-off between filler reinforcement and microstructural flaws when compared to the pristine epoxy. Although the mass density and hardness are efficiently increased by the nanoclay (as previously demonstrated), processing difficulties are also introduced.

A change in the resin's rheological behavior is suggested by the abrupt rise in void content at 5 wt%. Similar results in the literature indicate that the viscosity of the epoxy system grows exponentially as the concentration of high-aspect-ratio nanolayers rises above a critical threshold [19]. Higher levels of trapped air result from this increased viscosity, which prevents air bubbles from moving freely during the vacuum degassing stage.

Filler-induced porosity is a common occurrence in polymer nanocomposites, according to studies [19–21], which are consistent with the observed trend. Nanoclay agglomeration is more likely at greater weight fractions (e.g., 5 wt%). Because the resin may not completely penetrate the interstitial spaces between closely spaced clay platelets, these clusters serve as locations for the creation of micro-voids. The doubling of the void content at 5 wt% is a warning sign, even though the initial hardness data shows that the stiffening impact of the nanoclay is predominant. Despite the increases in surface hardness, these voids may serve as stress concentrators in structural applications, which could result in early fracture initiation and a decrease in ultimate tensile strength. As a result, the 3 wt% loading seems to be the ideal concentration for this system, offering substantial reinforcement while keeping the void content below the 0.6% cut-off.

Effect of nanoclay loading on hardness of epoxy composites

The epoxy nanocomposite's Shore D hardness increases with nanoclay addition from 0% to 3 % by weight, as seen in Figure 2. Pristine epoxy has a hardness of 74 Shore D, increases to 79 at 1%, roughly 88 at 3%, and decreases to 83 at 5 wt%. The consistent increasing trend indicates that matrix stiffness is improved by nanoclay reinforcement. Higher loadings result in the narrowest error bars, indicating more uniform dispersion.

Nanoclay platelets exfoliate and interact with the polymer chains to improve hardness, which restricts mobility and increases load transfer. The plateau-like increase from 1% to 3% indicates proper reinforcement without saturation, despite agglomeration problems at 5 wt%. This is in line with nanocomposites, which exhibit declining benefits after achieving maximum mechanical advantages at low-to-moderate loadings (1–3 wt%). Joseph et al. [19] confirmed similar results, indicating that exfoliation confirmed by XRD data raised Shore D hardness from 81.6 to 84.5 with 1 wt% nanoclay in PMMA denture resin without increasing flexural strength.

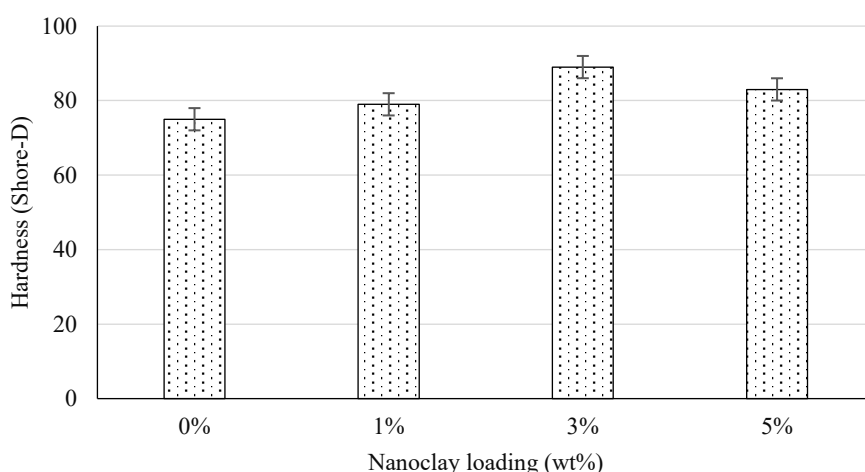


Figure 2. Hardness of epoxy - nanoclay composites.

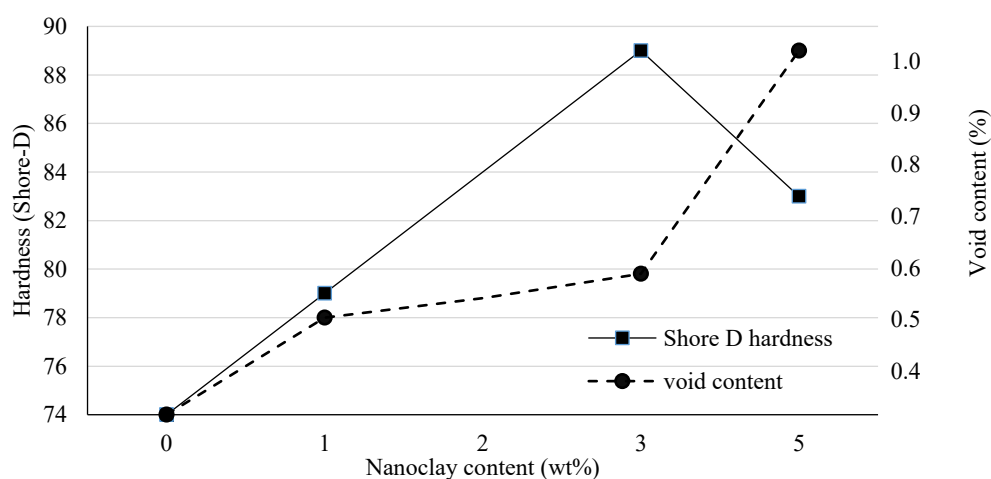


Figure 3. Correlation plot comparing the hardness and void content.

Figure 3 depicts the correlation plot comparing the hardness and void content. Shore D hardness increases gradually from the pure epoxy (74 SH) to the 3 wt% loading level, where it peaks at 89 SH. The successful exfoliation and dispersion of the high-stiffness silicate nanolayers is responsible for this notable increase (20%). This study's key outcome is that Shore D hardness dramatically dropped to 83 SH at the 5 wt% loading level after spiking at 89 SH at 3 wt%. There is a direct correlation between this decline in surface mechanical performance and a severe inflection in the void content, which rises to 1.017%. In the context of polymer nanocomposites, this kind of activity is a traditional sign that the critical filler concentration has been exceeded, at which point the negative consequences of microstructural flaws outweigh the silicate nanolayers' capacity to reinforce.

Two interconnected pathways are responsible for the mechanical deterioration shown at 5 wt% nanoclay loading. Increased void content first creates air-filled areas that lower the composite's effective load-bearing area and density, counteracting the nanoclays inherent stiffness and creating the appearance of softening. Second, too much filler causes tactoid development and nanoclay agglomeration, which break matrix integrity, impede stress transfer, and serve as stress concentrators. These results demonstrate that 3 wt% nanoclay is the ideal loading for maximum hardness while maintaining matrix quality, highlighting the importance of microstructural integrity minimizing voids and preventing agglomeration to mechanical performance. Therefore, a nanoclay composition of 3 wt% provides the ideal balance between mechanical reinforcement and structural integrity, even if 5 wt% yields the highest hardness.

Effect of Nanoclay Loading on Tensile Properties of Epoxy Composites

The tensile behavior of epoxy–nanoclay nanocomposites is depicted in Figure 4, where the modulus and tensile strength rise to 3 wt% nanoclay loading before decreasing at 5 wt%. This composition-dependent behavior is consistent with a shift from an exfoliated/intercalated morphology at low filler concentrations to a more aggregated system at higher contents. The discernible increase in tensile modulus at 1–3 wt% nanoclay can be explained by quantitative micromechanics models for platelet-reinforced composites. The Halpin–Tsai equation for longitudinal modulus (E_c) of composites with high aspect ratio fillers has been applied to epoxy-layered silicate systems on a regular basis:

$$E_c = E_m \frac{1+\xi\eta V_f}{1-\eta V_f} \quad (3)$$

where E_m is the matrix modulus, V_f is volume fraction, ξ is a shape parameter proportional to the platelet aspect ratio (L/t), and

$$\eta = \frac{\left(\frac{E_f}{E_m}\right)^{-1}}{\left(\frac{E_f}{E_m}\right)^{-1} + \xi} \quad (4)$$

Even at low volume fractions (1–3 wt%), values of $\xi \gg 1$ increase the modulus contribution since the effective aspect ratios of nanoclay platelets are typically between 100 and 1000. Therefore, the observed trend is consistent with the stiffening predicted by Halpin–Tsai under conditions of good platelet dispersion and interfacial adhesion. Comparable model–experiment agreement has been demonstrated by organoclay/epoxy composites in the 2–4% wt% range, with TEM imaging validating morphologies dominated by exfoliation [20, 21]. The deviation from Halpin–Tsai forecasts at 5 wt% is due to fillers entering an aggregation regime. Traditional micromechanics models struggle to effectively depict performance because platelet tactoids act as stress concentrators, reduce the accessible interfacial surface, and lower the effective aspect ratio conditions.

The increase in tensile strength up to 3 wt% nanoclay suggests effective stress transmission and fracture bridging. Nanoplatelets introduce processes that force crack deflection around clay platelets or tactoids and impede fracture propagation at the nanoscale, much as Orowan-type toughening. For platelets, this process increases the effective crack route length and local fracture energy. The mechanism can be approximated using the Orowan bowing relation:

$$\tau = \frac{Gb}{\lambda} \quad (5)$$

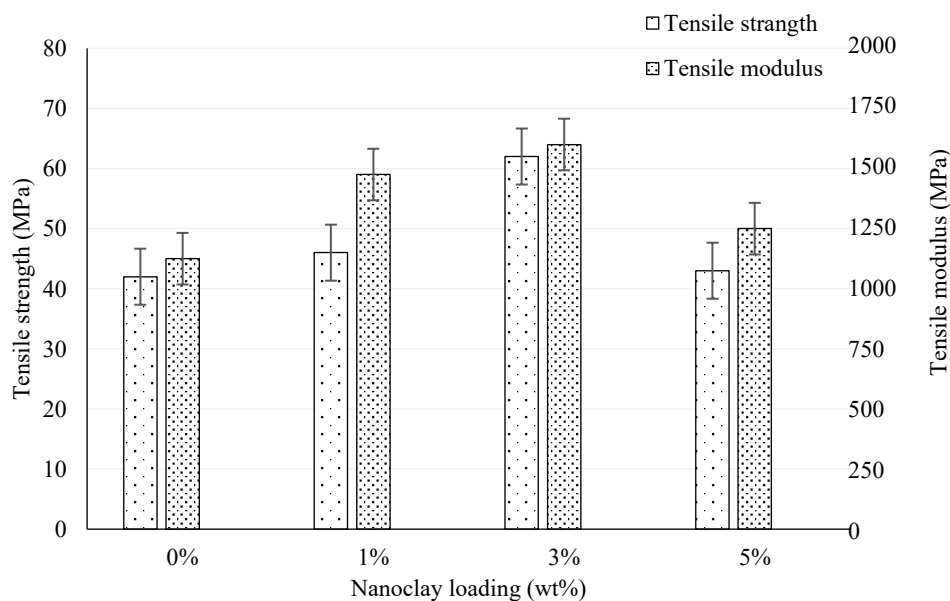


Figure 4. Tensile strength and tensile modulus of epoxy based nanoclay composites.

In this case, G stands for shear modulus, b for Burger's vector analog, and λ for platelet spacing. Here, b is Burger's vector analog, λ is platelet spacing, and G is shear modulus. Lower λ at moderate wt% leads to stronger strengthening until platelet aggregation at 5 wt% raises λ locally and creates voids or weak interfaces that dominate failure. Furthermore, as demonstrated in other epoxies nanoclay fracture studies [22–24], layered silicates operate as barrier tougheners by forcing crack points to repeatedly pass through platelet barriers, resulting in pull-out, microcracking, and crack bifurcation. Several processes combine to create toughness and strength before aggregation imposes decreasing returns.

Effect of nanoclay loading on impact strength of epoxy composites

The impact strength of epoxy nanocomposites with 0–5 wt% nanoclay is displayed in Figure 5. The impact strength of the neat epoxy is around $2.82 \text{ J}\cdot\text{m}^{-1}$. At 1 and 3 wt% loadings, the addition of nanoclay significantly increases impact resistance; at 3 wt%, the maximum value is $4.41 \text{ J}\cdot\text{m}^{-1}$, which is an improvement of almost 56% over plain epoxy. Impact strength decreases beyond this ideal filler amount, with the 5 wt% composite dropping to $3.22 \text{ J}\cdot\text{m}^{-1}$.

In total, the results demonstrate that impact strength is greatly increased by adding nanoclay up to 3 wt% before decreasing at higher loading. The greatest improvement is shown between 1 and 3 wt%, indicating that the composition with the highest energy absorption capacity is 3 wt%. The well-dispersed clay platelets that support energy-dissipative fracture mechanisms such as crack pinning, crack deflection, and localized plastic deformation within the epoxy matrix are responsible for this behavior. Platelet aggregation probably lessens these mechanisms by serving as stress concentrators at greater nanoclay contents (5 wt%), which lowers the material's resistance to crack propagation.

This pattern illustrates the interaction between the aggregation-driven embrittlement seen at greater clay concentration and energy dissipation mechanisms triggered by evenly distributed nanoclay platelets. Layered silicate nanoplatelets increase impact toughness at low to moderate clay levels through a variety of microstructural mechanisms, such as micro voiding, platelet pull-out, localized plastic shear, crack deflection and branching around platelets, crack pinning and bowing, like Orowan-type obstacles, and tortuous crack path formation due to tortuosity imposed by platelet geometry. Together, these processes raise fracture energy and prevent catastrophic crack propagation. For epoxy–organoclay systems, comparable improvements at 2–4 wt% clay have been extensively documented [25–27].

A reduction in impact strength at 5 wt% loading above 3 wt% nanoclay indicates a shift in fracture behavior. Increased viscosity during processing, which results in void formation and nanoclay agglomeration—which act as stress concentration sites and promote brittle fracture—explains this decline.

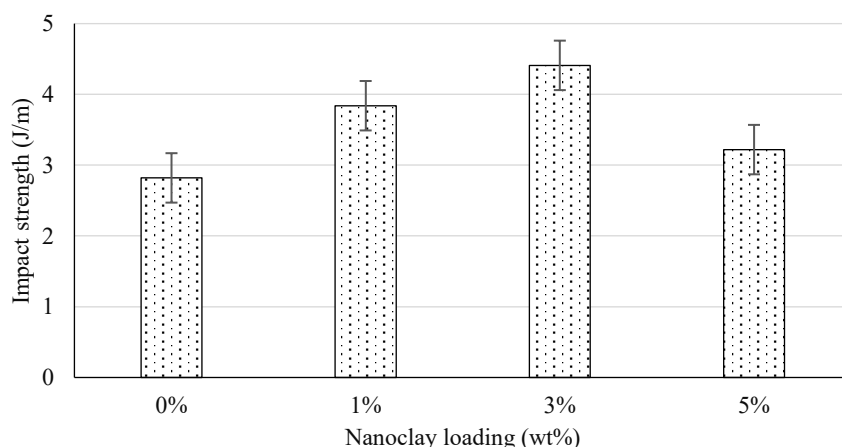


Figure 5. Impact strength of epoxy based nanoclay composites.

The change in slope between lower (0–1 wt.%), intermediate (1–3 wt.%), and higher (3–5 wt.%) nanoclay loadings suggests a change in the main fracture mechanisms from matrix-dominated fracture to nanofiller-assisted energy dissipation and eventually to agglomeration-induced premature failure.

A statistical analysis of the experimental data indicates that nanoclay loading has a considerable effect on the Izod impact strength of epoxy composites. The analysis of variance (ANOVA) performed at a 95% confidence level rejects the null hypothesis that all mean impact strength values are the same since the computed F-value is greater than the critical F-value. Furthermore, the post-hoc Tukey's multiple comparison test indicates that the 3 wt% nanoclay composition is statistically superior to the other compositions in terms of impact energy absorption. This illustrates that epoxy composites have an optimal nanoclay loading that improves impact resistance.

CONCLUSIONS

- The most appropriate filler concentration for dependable epoxy composite performance is 3 wt%, even if a greater nanoclay content (>5 wt%) further improves hardness. However, the corresponding fast increase in void content suggests decreased processability and possible structural degradation.
- The use of nanoclay enhances the tensile strength, and modulus of epoxy composites up to 3 wt% as a result of efficient platelet dispersion and polymer-filler interactions.
- The stiffening behavior corresponds with Halpin–Tsai micromechanics predictions for high aspect-ratio nanoplatelets in exfoliated or intercalated morphologies.
- Impact strength enhanced by about 56 at 3 wt% due to energy-dissipative fracture mechanisms, including crack deflection, crack pinning, platelet pull-out, and localized plastic shear.
- Property degradation at 5 wt% nanoclay is ascribed to tactoid aggregation, void formation, stress concentrations, and reduced interfacial area.
- ANOVA and Tukey's post-hoc analysis validated that 3 wt% nanoclay yields statistically improved mechanical performance, signifying an appropriate filler loading for epoxy nanocomposites.

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