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## **Effect of Nano Silica Fillers on Dynamic Mechanical Performance and Accelerated Ageing Behavior of Carbon/Epoxy Composites**

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**Abstract:** The impact of nano-silica (NS) fillers on the mechanical and aging characteristics of carbon fiber epoxy composites (CFECs) is examined in this work. Composite samples with 0–6 weight percent NS were exposed to UV light and moisture to hasten aging. According to Dynamic Mechanical Analysis (DMA), the glass transition temperature ( $T_g$ ) improved by 6.6%, reaching a peak of 48.57 °C, while the storage modulus ( $E'$ ) increased by up to 27.5%, from 184 GPa in NS0-U to 234.5 GPa in NS4-U. At 2–4% filler loading, interlaminar shear strength (ILSS) decreased by 45% as a result of nanosilica addition. The NS4-U sample's Charpy impact strength was 60 kJ/m<sup>2</sup>, yet there was a noticeable overall drop in impact resistance as the filler content increased. Upon ageing, all properties showed degradation; however, NS4-A composites retained higher mechanical properties than the aged unfilled baseline, confirming the filler's role in enhancing environmental resistance. These results highlight that optimal NS loading (4 wt%) can significantly reinforce CFECs while offering improved ageing durability.

**Keywords:** Composite weathering, Nanosilica fillers, Composite mechanical properties, Composite life deterioration

### **Introduction**

Carbon fiber-reinforced epoxy composites (CFEC) are extensively used across industries such as aerospace, automotive, and advanced structural engineering due to their high specific strength, exceptional stiffness-to-weight ratio, excellent corrosion resistance, and favorable fatigue performance. These properties make CFEC an ideal material for applications that demand high mechanical strength without adding significant weight. However, a major drawback of composite materials is their susceptibility to long-term degradation. Environmental exposure can lead to polymer matrix deterioration primarily through processes like post-curing or polymer chain scission, ultimately compromising structural integrity and leading to in-service failures. Ensuring the long-term

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environmental stability of CFEC remains a significant challenge in extending their reliability and performance in demanding conditions.

Environmental factors can negatively impact the polymer matrix, where they induce degradation mechanisms within the matrix. The main factors are moisture, oxidative agents, thermal fluctuations, and ultraviolet (UV) radiation. The exposure to UV radiation leads to oxidation and chain cleavage of the epoxy network. While the absorption of moisture causes plasticization, separation the fiber interface from the matrix, and hydrolysis (Ci et al., 2025; Oliveira et al., 2022). These key factors have a combined impact weakening the fiber-matrix interface, fragmentation of the matrix, and deterioration of mechanical properties such as dynamic modulus, impact toughness, and interlaminar shear strength (ILSS) (Boubakri et al., 2010; Brown & Greenwood, 2002; Cysne Barbosa et al., 2017; Kumar et al., 2002; Siriruk & Penumadu, 2014; Springer, 1994). These degradation disciplines indicate risks to the the integrity of CFCs structures over time that leads to raise the concerns for long-term applications.

Numerous studies have investigated the behavior of carbon fiber-reinforced epoxy composites (CFECs) under various environmental conditions such as temperature fluctuations, humidity, and moisture absorption. However, comparatively fewer studies have focused on developing effective mitigation strategies to enhance the environmental durability of CFEC. One promising route involves enhancing the resin matrix through the addition of inorganic nanofillers such as nano-silica, nanoclays, and alumina. Inorganic fillers have been shown to improve matrix toughness, reduce permeability, and enhance the overall mechanical performance of fiber-reinforced composites. In particular, the incorporation of glass microparticles as secondary reinforcements has been reported to significantly enhance the flexural behavior of composite laminate (Farsani et al., 2013) , while silica-modified epoxy matrices exhibited reduced water uptake and increased stiffness (Chang et al., 2013). Another study reported a similar trend, where the incorporation of nanofillers led to improvements in both impact strength and fracture toughness, while also enhancing resistance to moisture ingress (Yousif & Haddad, 2013). Theoretical frameworks support these findings by highlighting mechanisms such as crack deflection, filler-induced toughening, and enhanced interfacial interactions (Awaja et al., 2016).

Despite recent advances in CFECs, the influences of nanosilica additives and environmental aging on the mechanical performance are still poorly understood, and need more studies. So, there is a critical research gap, especially in advanced applications that require environmental reliability and mechanical durability as main requirements. To fill this gap, the current work under the conditions of accelerated degradation/aging simulates environmental deterioration by studying the mechanical behavior of CFECs reinforced with nanosilica. The mechanical properties of designed composites were evaluated before aging and after aging. Interlaminar shear strength, Charpy impact strength, and dynamic mechanical properties were the main mechanical properties incuded. The findings aim to inform resin matrix modification strategies that enhance the long-term durability and reliability of CFECs in challenging service environments. Moreover, by examining the correlation between nano-silica reinforcement and environmental resistance, this study contributes to the development of next-generation CFEC systems engineered for sustained performance under harsh operational conditions.

## Materials and Methods

CFRP composites were fabricated using woven carbon fabric with a twill weave pattern and an areal density of 200 g/m<sup>2</sup> as the reinforcement phase. Epikote 816 epoxy resin was used as the matrix, while nano-silica particles (sized between 20–30 nm) sourced from USA Research Nanomaterials were included as the inorganic filler reinforcement. Composite laminates were manufactured using the vacuum bagging method, incorporating different filler loadings of 0%, 2%, 4%, and 6% by weight, as outlined in Table 1.

For filler inclusion, the required amount of nano-silica was dispersed into a pre-measured volume of epoxy resin and subjected to ultrasonic agitation for 60 minutes to achieve uniform distribution. The layup consisted of eight layers of 300 mm × 300 mm carbon fabric arranged in a [0/90]<sub>4s</sub> stacking sequence. Resin was applied through hand lay-up, and vacuum pressure was subsequently applied to eliminate excess resin and ensure consolidation. In the case of filler-modified composites, an additional sonication step using a Hawsin Powersonic 420 unit was carried out for 40 minutes after filler-resin mixing to further enhance dispersion before impregnation onto the fabric layers. The composite sample preparation setup is shown in Figure 1. The fabricated laminates were cured at ambient conditions for 24 hours and then sectioned into standard specimen sizes appropriate for each mechanical test. Following fabrication, the samples underwent accelerated environmental aging, sample nomenclature and details are mentioned in Table 2.



Figure 1. Composite fabrication setup

Table 1. Composite design matrix

Parameter	Units	Values			
Filler content	wt %	0	2	4	6
Fiber Volume Fraction ( $V_f$ )	-	0.60			
Composite Aging	-	Unaged		Aged	

Table 2. Nomenclature of the sample used in the current study

S.No	Filler	Filler wt%	Aging Condition
NS0-U		0	
NS2-U		2	
NS4-U		4	Unaged
NS6-U		6	
NS0-A	Nano silica	0	
NS2-A		2	
NS4-A		4	Aged
NS6-A		6	

### Accelerated Ageing

Accelerated environmental ageing of the composite samples was performed using the Xenon Lab 325E chamber (Mesdan Lab), which replicates real-world outdoor conditions such as solar radiation, humidity, and thermal exposure. During testing, the chamber operated at an irradiance level of 60 W/m<sup>2</sup>, with relative humidity maintained at 50%. The temperature inside the chamber was controlled automatically according to preset aging conditions at (65-70 °C). Each composite specimen was subjected to a total exposure duration of 7 hours, in accordance with the guidelines of ASTM G154. This standard specifies various exposure cycles involving controlled irradiance and moisture conditions, and allows for customized cycles within a typical range of 4 to 8 hours, depending on the intended simulation of environmental conditions.

### Characterization of Composites

In compliance with ASTM D4065, dynamic mechanical analysis (DMA) was performed to assess the composites' temperature-dependent viscoelastic behavior. For testing, rectangular specimens with dimensions of 60 mm × 13 mm × 2 mm were made. A TA Instruments DMA Q-800 (New Castle, DE, USA) in three-point bending mode was used to evaluate the dynamic mechanical response of DMA samples. In order to prevent oxidative deterioration, tests were carried out in a nitrogen atmosphere with a frequency of 1 Hz and a heating rate of 3 °C/min over a temperature range of 25 – 100 °C.

In accordance with ASTM D2344, the short beam shear (SBS) test was used to assess the composite specimens' interlaminar shear strength (ILSS). A compressive load was applied during the test at a steady crosshead speed of 1.3 mm/min until the first failure occurred. For every specimen, the maximum load at the point of failure was noted. Three specimens were evaluated for each type of material, and the average ILSS value was determined. The ILSS was computed using the following equation:

$$\tau = \frac{3 \cdot P_R}{4 \cdot b \cdot h} \quad (1)$$

$\tau$  = apparent Interlaminar shear strength (MPa);  
 $P_R$  = maximum load at the moment of first failure (N);  
 $b$  = width of the specimen (mm);  
 $h$  = thickness of specimen (mm).

Charpy impact strength of the composite specimens was evaluated in accordance with the ISO 179 standard, which aligns with the guidelines provided in ASTM D256. Test specimens were prepared with dimensions of 80 mm  $\times$  10 mm, and three samples were tested for each composite type. The impact tests were performed using a Zwick Roell HTI5P impact testing machine. For consistency and reliability, the mean value of the three measurements was taken as the representative impact strength for each sample group.

## Results and Discussion

### Dynamic Mechanical Properties

The influence of nano-silica fillers and accelerated ageing on the viscoelastic properties of carbon fiber epoxy composites was investigated using dynamic mechanical analysis (DMA) in accordance with ASTM D4065. Key parameters such as storage modulus ( $E'$ ), loss modulus ( $E''$ ), damping factor ( $\tan \delta$ ), and glass transition temperature ( $T_g$ ) were evaluated. Figures 1 to 3 illustrate the temperature-dependent trends of these properties for both aged and unaged samples.

### Storage Modulus

The storage modulus ( $E'$ ), indicative of the material's stiffness, showed a strong dependence on both filler content and ageing conditions. As illustrated in Figure 1 (a), unaged composites with nano-silica fillers exhibited a substantial increase in  $E'$  compared to the baseline unfilled composite (NS0-U). Notably, NS4-U and NS6-U samples demonstrated the highest stiffness among the unaged specimens, indicating effective reinforcement at these filler loadings.

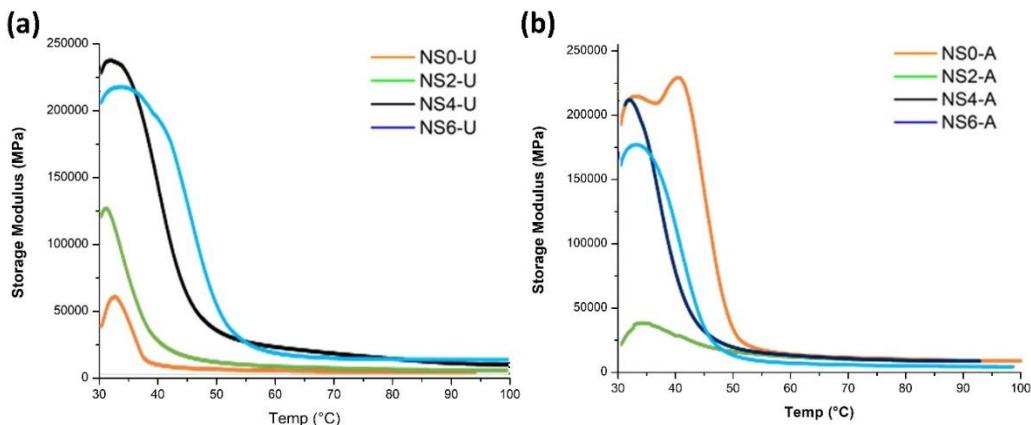


Figure 1. Storage modulus graphs of composites; (a) Unaged, and (b) Aged

Conversely, ageing had a detrimental effect on stiffness, as seen in Figure 1 (b). Most aged samples exhibited a reduction in  $E'$ , except NS4-A, which retained moderate stiffness. The decline in modulus post-ageing is attributed

to microstructural deterioration, such as interfacial debonding, microcrack formation, and plasticization of the matrix due to moisture ingress and UV exposure (Awaja et al., 2016; Le Guen-Geffroy et al., 2019).

### Loss Modulus

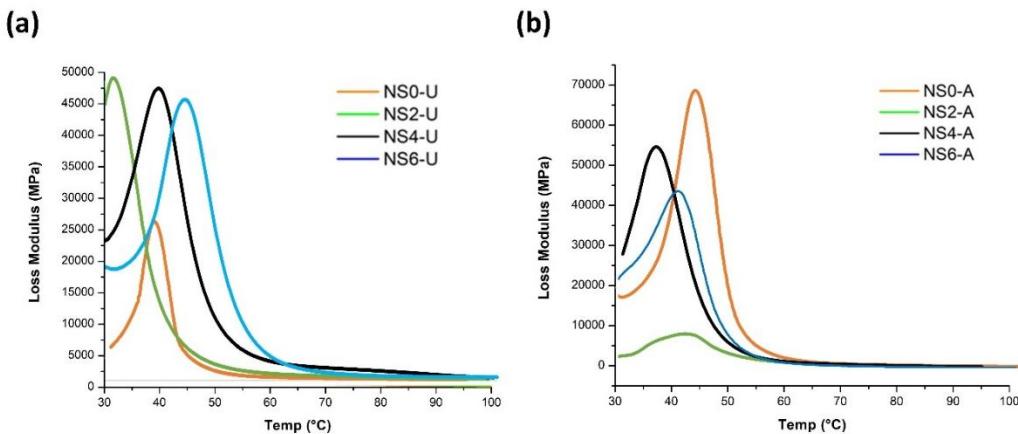


Figure 2. Loss modulus graph of composites; (a) Unaged, and (b) Aged

The loss modulus represents the material's ability to dissipate energy through internal friction, making it a key indicator of viscoelastic damping behavior. In this study, the incorporation of nano-silica into carbon/epoxy composites was found to significantly influence the loss modulus, especially in the unaged condition.

As shown in Figure 2 (a) In comparison to the NS0-U composite, the inclusion of nanosilica increased the loss modulus values for all of the nanosilica-loaded composites. This decline is explained by the stiffening impact of the nano silica particles, which lowers the polymer chains' mobility and restricts their capacity to experience large-scale deformations, hence lowering the loss modulus. (Klepka et al., 2021).

In contrast, as shown in Figure 2 (b), the aged composites have lower loss modulus values, especially in the samples that were filled with nanosilica. Degradation and matrix embrittlement at the fiber–matrix interface are responsible for this decrease since they restrict the material's capacity to release energy through viscoelastic processes. It's interesting to note that the unfilled aged composite (NS0-A) displayed a comparatively larger loss modulus. This could be because of enhanced chain mobility brought on by polymer plasticization and the development of microcracks as it ages (Guo et al., 2021).

In conclusion, the damping behavior of the composites in their unaged state was enhanced by nano-silica fillers. Their presence, however, seemed to inhibit the rise in molecular mobility brought on by aging, which resulted in decreased loss modulus values following exposure to the environment. This implies that, while at the price of a decreased damping ability, nano-silica may aid in maintaining composite rigidity as it ages.

### Tan Delta

The damping behavior of the composites, assessed through  $\tan \delta$ , is depicted in Figure 3. In the unaged condition, Figure 3(a), most nano-silica-filled composites exhibited lower  $\tan \delta$  values compared to the unfilled reference (NS0-U), except for NS2-U, which showed a slight increase. This suggests that higher nano-silica contents (4% and 6%) restrict polymer chain mobility and reduce energy dissipation, resulting in a stiffer, more elastic composite behavior. This suggests that the composites with nanosilica fillers are more effective in absorbing energy during cyclic loading, which can be attributed to the enhanced interfacial adhesion between the matrix and the fillers (Wang et al., 2002).

In contrast, the elevated  $\tan \delta$  of NS2-U implies enhanced energy absorption during cyclic loading, likely due to optimal filler dispersion and improved interfacial adhesion at lower filler concentrations. Among the aged samples Figure 3 (b), NS6-A recorded the highest  $\tan \delta$ , indicating that at higher filler content, nano-silica may help retain or improve damping capacity even after ageing, possibly due to preserved interfacial integrity and stress relaxation mechanisms.

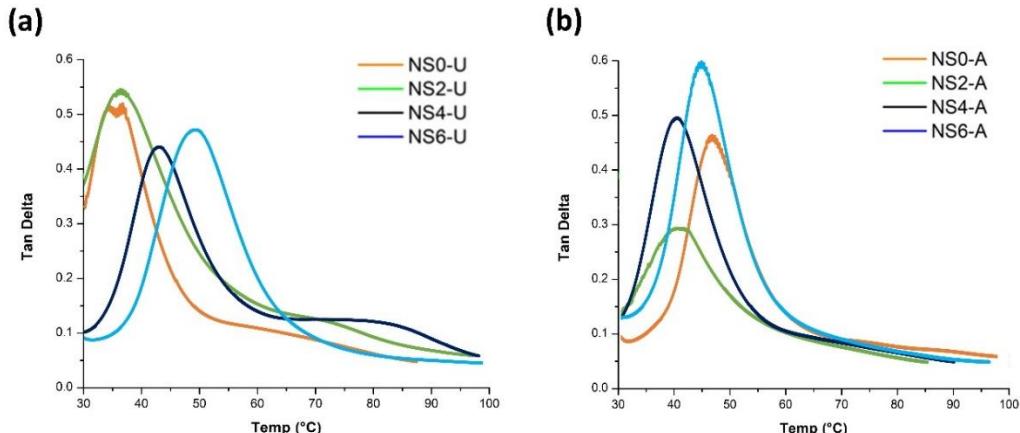


Figure 3. Tan delta graphs of composites; (a) Unaged, and (b) Aged

### Impact Strength

As seen in Figure 4, the impact test was used to assess the impact behavior of NS-loaded CFRP. Because the addition of nanosilica particles results in a poor matrix reinforcing interface, which causes the composite to fracture under impact force, the impact strength of composite materials was reduced. The NS4-U (60 KJ/m<sup>2</sup>) composites showed the highest impact strength for NS-loaded CFRP. The capacity of nano-silica to prevent crack initiation and propagation through particle-matrix debonding processes, energy dissipation through plastic deformation, and crack deflection is frequently cited as the reason for this improvement (Chikhi et al., 2002). However, as seen in the case of NS4-U, the propensity for nanoparticle agglomeration at greater filler concentrations might result in stress concentration sites, encouraging early failure and lowering the impact performance.

Following accelerated ageing, the composites exhibited a noticeable decrease in impact resistance, indicating increased brittleness due to matrix embrittlement and degradation at the fiber–matrix interface. This degradation is commonly associated with thermo-oxidative ageing, which reduces chain mobility, accelerates microcrack formation, and weakens interfacial bonding (Johnsen et al., 2007). Additionally, moisture ingress during environmental exposure may further plasticize or hydrolyze the matrix, amplifying the loss in toughness (Xu et al., 2024). Nevertheless, in some aged samples, an increase in recorded impact strength was observed, potentially resulting from residual curing effects or stress relaxation that alter crack propagation dynamics by temporarily enhancing cross-linking density or reducing internal stresses (Shaker et al., 2024).

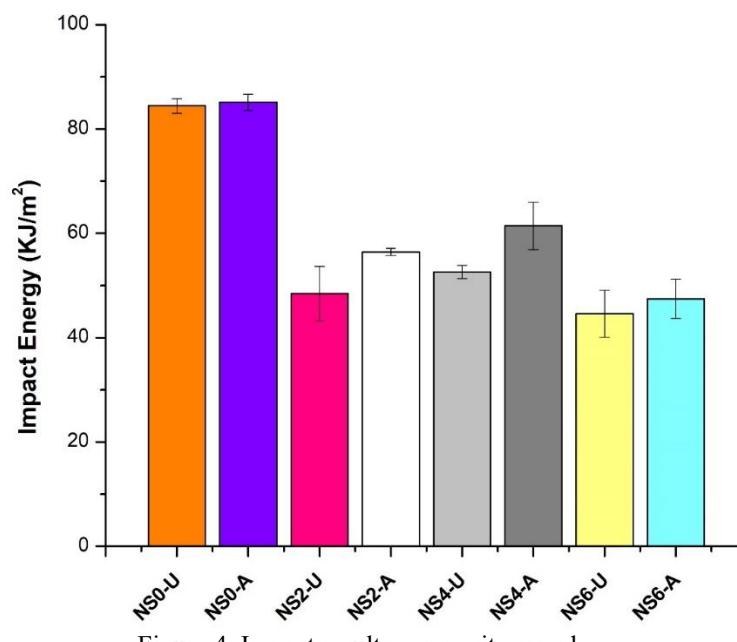


Figure 4. Impact results composite samples

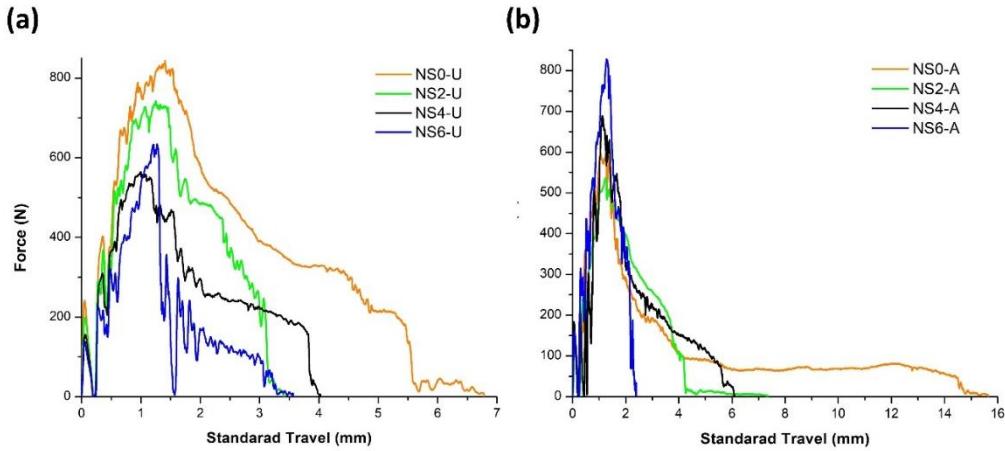


Figure 5. Force v.s standard travel graphs; (a) Unaged (b) Aged

The corresponding force versus standard travel curves for both aged and unaged nano-silica-filled composites are presented in Figure 5, offering deeper insight into their fracture response under impact loading. The incorporation of nano-silica particles led to a reduction in both peak force resistance and overall toughness of the composites in the unaged condition. Notably, the reference sample (NS0-U) exhibited the highest peak force and absorbed the maximum energy, as indicated by the largest area under the curve in Figure 5 (a). In contrast, an opposite trend was observed for the aged composites, as shown in Figure 5 (b). Nano-silica-loaded composites demonstrated higher resistance to force compared to the aged reference (NS0-A), although a general reduction in toughness was still evident due to aging-induced matrix embrittlement. Among the aged samples, NS6-A exhibited the highest peak force, followed by NS4-A, indicating that nano-silica addition may mitigate some of the adverse effects of aging. This behavior aligns with previous studies suggesting that silica nanoparticles can enhance the thermal stability and aging resistance of polymer matrices by restricting molecular mobility and acting as physical crosslinking sites (Carrasco & Pagès, 2008). Consequently, the presence of nanoparticles likely delays crack initiation and reinforces the composite against degradation-induced weakening.

#### Interlaminar Shear Strength (ILSS)

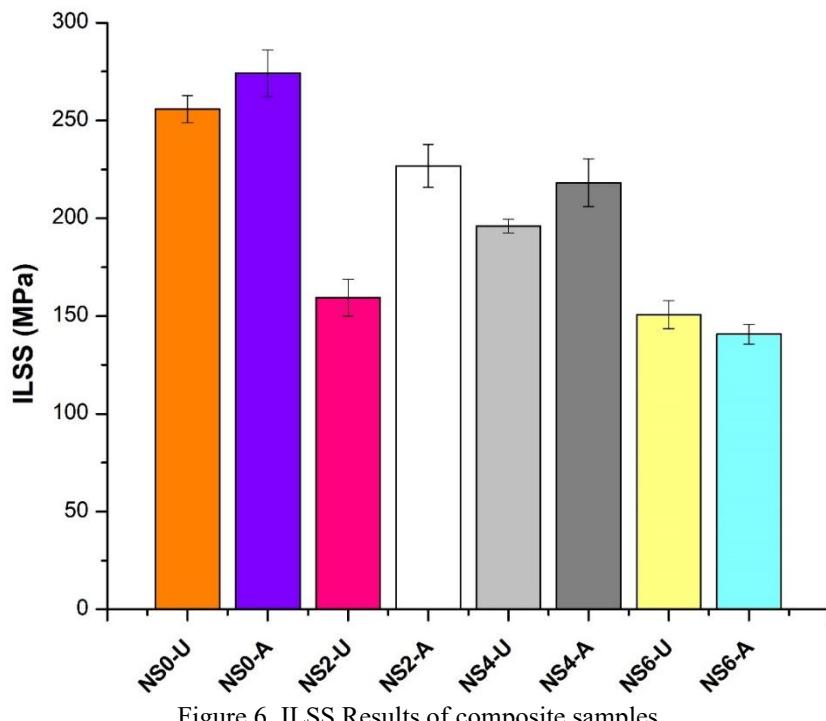


Figure 6. ILSS Results of composite samples

As shown in Figure 6, the incorporation of nano-silica particles into the epoxy matrix demonstrated a negative influence on interlaminar shear strength (ILSS), particularly at lower filler concentrations. The deterioration in ILSS is attributed to the poor fiber–matrix interfacial bonding, promoted by the inadequate dispersion of nano-silica particles, which likely reduces the mechanical interlocking at the interface. Gojny et al reported a similar trend that at lower nanofiller (CNTs), poor dispersion resulted in decreased interfacial properties due to agglomeration and inadequate stress transfer (Gojny et al., 2005). The data suggest that an optimal filler loading exists in the range of 2–4 wt%, where the composites exhibit the most significant improvements in ILSS. Beyond this range, particularly at 6 wt%, a decline in ILSS was observed. This reduction may be due to filler agglomeration and the formation of microvoids or weak interfacial regions, which can act as stress concentrators and compromise the integrity of the fiber–matrix interface. These findings highlight the importance of controlled nanoparticle dispersion to maximize interfacial strength without introducing processing-induced defects.

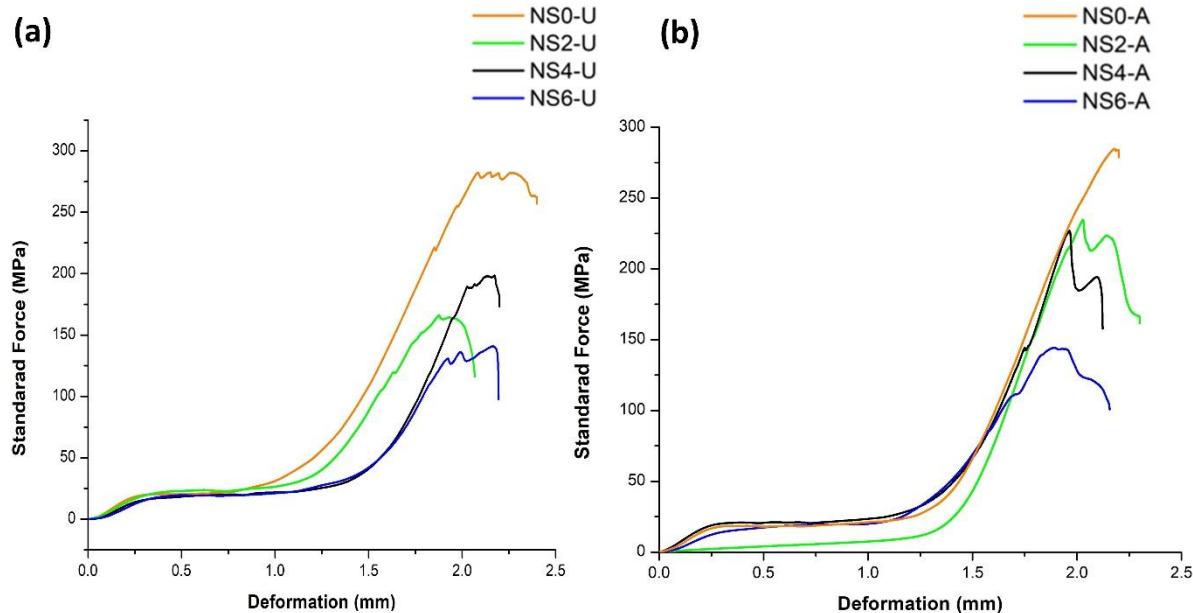


Figure 7. Force vs displacement graphs: (a) Unaged, and (b) Aged

The Force vs. Deformation curves derived from the ILSS tests are presented in Figure 7 for both unaged and aged composite samples. As shown in Figure 7 (a), the NS0-U exhibited the highest peak force (~290 MPa), showing a superior interfacial bonding and load-bearing capability. Incorporation of nanosilica particles NS2-U and NS4-U shows a decline in maximum force, with NS4-U having the highest force resistance to fracture. The ILSS test findings are consistent with other testing that 6 wt% addition of particles leads to poor mechanical properties, mainly due to poor stress transfer.

For the aged specimens, Figure 7 (b) reveals that all composites showed reduced force resistance capacity due to aging compared to unaged counterparts. NS0-A retained highest peak force (~270 MPa) though slightly reduced compared to NS0-U. Among reinforced particles NS4-A showed better retention of force capacity (~240 MPa) to NS2-A and NS6-A indicating better resistance to accelerated aging. NS6-A again exhibited the weakest performance, affirming that higher filler loading leads to water ingress, plasticization, and microstructural defects, further deteriorating mechanical performance under environmental stress.

## Conclusion

This study investigated the influence of silica nanoparticle reinforcement and accelerated ageing on the dynamic mechanical performance, impact resistance, and interlaminar shear strength (ILSS) of woven carbon fiber-reinforced epoxy composites. The results indicate that moderate loading of nanosilica particles (4 wt%) significantly improved storage modulus and glass transition properties in unaged samples, indicating enhanced stiffness and thermal resistance. However higher loading percentage led to deterioration in mechanical and thermal properties due to agglomeration and poor dispersion.

Mechanical properties for nanosilica loaded CFRPs increased at intermediaite concentrations, but the overall trend showed reduction attributable to increased brittleness and stress concentrators. The NS4-U showed best trade-off between toughness and reinforcement with a higher level of reduced impact and interlaminar strength. Accelerated ageing had a pronounced degrading effect across all formulations, reducing both E' and Tg, and contributing to matrix embrittlement. Despite this, nano-silica-loaded composites, particularly those with 4 wt% retained relatively higher mechanical performance than the aged baseline composite (BA), demonstrating greater environmental durability.

In summary, the findings suggest that nano-silica incorporation at optimal levels (4 wt%) can enhance the stiffness and thermal performance of carbon/epoxy composites while providing moderate resistance to environmental ageing. However, trade-offs in impact toughness and interlaminar shear strength must be carefully considered during composite formulation. These insights offer valuable guidance for the design and optimization of multifunctional carbon fiber composites intended for applications where both mechanical robustness and long-term environmental resistance are critical. Future research could explore the long-term durability of nano-silica-modified composites under real-world environmental conditions. Such enhancements hold promise for lightweight, impact-resistant components in automotive and aerospace applications.

## Scientific Ethics Declaration

\* The authors declares that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

## Conflict of Interest

\* The authors declare that they have no conflicts of interest

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