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Research Article

Experimental Investigation on the Tensile Behavior of MWCNT – Nano Silica Epoxy Hybrid Nanocomposites

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ABSTRACT

This study aimed to investigate the mechanical properties of epoxy nanocomposites filled with single and binary combination of multiwalled carbon nanotube (MWCNT)-nano silica (NS) nanoparticles. The epoxy nanocomposites were produced using the mold casting method, and different filler ratios were employed to create nanocomposite samples. For the single particle-filled samples, filler ratios of 0.1, 0.3, 0.5, and 0.7 wt.% for MWCNT and 0.5, 1.0, and 1.5 wt.% for NS were used. Additionally, hybrid samples were produced using filler ratios of 1:1, 1:2, and 1:3 (MWCNT: NS). The tensile test results indicated notable enhancements in the tensile behavior of the nanocomposite, attributed to the integration of nanoparticles into the epoxy matrix. Particularly, tensile strength values improved by 28.35% and 21.25% in C3 (0.5 wt.% MWCNT) and S2 (1.0 wt.% NS) composite samples compared to the pure sample, respectively. Additionally, the hybrid nanoparticle-filled composite samples introduced a synergistic effect on the tensile behavior of the nanocomposite. Especially, the hybrid sample H1 (1:1) showed the maximum enhancement in tensile strength by 44.26%. Significant improvements were also observed in tensile strain values. Compared to the control sample, the maximum improvement was recorded as 143% in the H2 hybrid sample (1:2).

1. Introduction

Epoxy, a prominent thermosetting polymer, has garnered considerable attention in recent years owing to its exceptional properties, including elevated tensile strength, minimal shrinkage during the curing process, commendable chemical and thermal resistance, and superior adhesion. Despite these favorable attributes, the inherent brittleness of epoxy polymers poses a significant drawback, characterized by susceptibility to crack initiation and propagation [1–3]. This brittleness is caused by the presence of microcracks, and micro voids induced by polymerization, which limits their effectiveness in high-performance applications, especially in aerospace structures [4–6].

Over the past decade, there has been a growing significance attributed to epoxy nanocomposites, wherein epoxies are augmented with micro/nano-sized particles, owing to their exceptional properties. The large surface area of the incorporated nanoparticles confers new macroscopic properties such as improved mechanical hardness and enhanced fracture toughness [7–11].

Seloğlu et al. [12] conducted a comparative study on the effect of the individual addition of multi-walled carbon nanotube (MWCNT), nano silica (NS) and nano-ZnO (NZ) on the mechanical properties of geopolymer mortar composites. In comparison to the control samples, the study reports that the mechanical strength of all samples was found to be significantly enhanced by the addition of individual nanomaterials. Among the various nanomaterials tested, the samples containing MWCNT exhibited the highest levels of compressive and flexural strength. In addition, geopolymer mortar samples containing NS and NZ also showed significant improvements in strength, with samples containing NS performing better than those containing NZ. In another study, Zappalorto et al. [13] examined the mechanical response of an epoxy/silica nanocomposite system. They conducted tensile and fracture tests and analyzed the results. Additionally, the researchers investigated the impact of the curing procedure on the mechanical properties of the nanocomposites, using two different curing conditions. Their results show that the fracture toughness epoxy is enhanced by the introduction of nanoparticles. Depending on the curing process, the

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strength values of the samples either increased or decreased. Kumar et al. [14], compared the tensile and flexural properties of nano SiO_2/ epoxy polymer nanocomposites. They carried out tensile and 3-point bending tests on nanocomposites featuring varying SiO_2 contents of 2, 4, 6, and 8 wt.%. Their findings indicated that the mechanical behaviors of SiO_2/epoxy nanocomposites exhibited improvement with the dispersion of fillers, particularly up to 4 wt.% of SiO_2 nanoparticles. However, beyond the 4 wt.%, there was a decline in mechanical properties. This deterioration was attributed to a substantial increase in the agglomeration and sedimentation of SiO_2 nanoparticles during the extended curing time. Specifically, a 4 wt.% SiO_2 dispersion led to increases in tensile strength of 30.57% increase, flexural strength of 17%, and flexural modulus of a remarkable 76%. Kaybal et al. [15] explored the distribution of nano SiO_2 and its impact on mechanical properties. They produced nanocomposites with varying weight percentages of SiO_2, ranging up to 5% by weight. The test results revealed that the optimal tensile and bending strength values were achieved when incorporating 3% by weight of nano SiO_2 into epoxy nanocomposites. It has been reported that beyond this concentration particle agglomeration commenced, leading to an adverse on the mechanical properties of the nanocomposite. In a related investigation, Chen et al. [16]examined the mechanical behaviors of epoxy composites incorporating nano silica with minimal agglomeration. They observed substantial enhancements in tensile modulus and fracture toughness for nanocomposites featuring filler content below 10%.

Because of their exceptional mechanical strength, high heat conductivity, distinctive microstructure, and resistance to corrosion, carbon nanotubes (CNT) have been the subject of several studies [17–19]. Bansal et al. [18] conducted a comprehensive study to investigate the mechanical effects associated with the introduction of very small concentrations of CNT as reinforcement. Remarkably, even with a minimal CNT reinforcement of 0.25 wt.%, the epoxy-CNT composite exhibited significant improvements in both elastic modulus and hardness values. Specifically, the elastic modulus increased from 2.87 GPa to 3.93 GPa, and hardness values rose from 0.208 GPa to 0.242 GPa. The outcomes of this study underscore the positive influence of even modest CNT reinforcements on the mechanical properties of the composite material. Gantayat et al [19] conducted an analysis of the mechanical properties of an epoxy resin polymer matrix through the incorporation of varying weight percentages (0.4, 0.6, and 1.0 wt.%) of MWCNT. Preceding their inclusion, MWCNT underwent a chemical treatment involving mixed acid for functionalization. This functionalization process effectively disrupted the atomic forces between the nanotubes, mitigating their tendency to agglomerate. The tensile strength and modulus of the epoxy experienced noteworthy enhancements attributed to the good dispersion of functionalized MWCNT within the epoxy polymer and the establishment of robust interfacial adhesion between the epoxy matrix and MWCNT. The strength gradually increased and reached its maximum enhancement of 27% at 0.6 wt.% of functionalized MWCNT inclusion. Similarly, tensile modulus displayed a maximum enhancement of 14% under the same filler content. Montazeri et al. [20] studied the mechanical properties of a composite, examining the impact of both untreated and acid-treated MWCNT. The study revealed that the incorporation of MWCNT at specific weight ratios led to notable enhancements in the mechanical properties of the composite. Furthermore, their another study [21] concentrated on both the mechanical and viscoelastic effects resulting from the addition of MWCNT to epoxy resin at varying weight ratios (0, 0.1, 0.5, 1, and 2 wt.%), demonstrating an overall improvement in the viscoelastic properties of the nanocomposite. Specifically, the investigation highlighted that the concentration of 0.5 wt.% MWCNT in epoxy resin yielded the most significant enhancement in viscoelastic properties when compared to both the pristine epoxy and other nanocomposite formulations. Salman et al. [22] fabricated nanocomposites through the casting method, incorporating single-walled carbon nanotubes (SWCNT) into resin matrices at varying weight concentrations (0.1, 0.3, 0.5, and 1 wt.%). Numerous mechanical tests, including tensile, flexure, and hardness tests, were conducted on the nanocomposites. Additionally, scanning electron microscope (SEM) micrographs illustrated the homogeneous dispersion of SWCNT within the epoxy. Furthermore, Raman spectroscopy and x-ray diffraction (XRD) results corroborated the findings from SEM. The research elucidated enhancements in mechanical properties attributable to the uniform distribution of SWCNT within the epoxy matrix. Ultimately, there were notable increases in elastic modulus, tensile strength, flexural strength, and hardness values. Gojny et al. [23] examined the mechanical properties of epoxy nanocomposites featuring distinct types of CNT at filler ratios of 0.1, 0.3, and 0.5 wt.%. The investigation encompassed SWCNT, Double-Walled CNT (DWCNT), MWCNT, and amino-functionalized DWCNT. The findings of the study assert that the incorporation of CNT leads to enhancements in the mechanical properties of the nanocomposites.

Researchers have also shown significant interest in hybridized nanoparticle filled composites due to their exceptional mechanical, electrical, and thermal attributes. A study conducted by Ismail et al. [24] investigated the impact of MWCNT and SiO_2 particles on the mechanical properties of natural rubber nanocomposites. The study found that MWCNT/SiO_2 hybrids showed improvements in the mechanical performance of the nanocomposite, such as increased tensile strength, and tensile modulus. This observation underscores the advantageous effects of combining diverse nanoparticle materials. A comprehensive comparative analysis of all mixtures elucidated that hybrid matrices outperformed their single filler counterparts across various parameters, including tensile strength, fracture mechanics, electrical conductivity, and thermo-mechanical properties [25].

Based on the literature review, although the effect of single and binary combinations of MWCNT and NS nanofillers on the tensile properties of rubber nanocomposites has been studied, it appears that there is no detailed experimental study on its effect on the mechanical performance of commercially widely used epoxy nanocomposites. This study aims to assess the effect of utilizing binary (hybrid) and single-phase MWCNT and NS particles on the tensile characteristics of composites. For this purpose, the epoxy resin was mixture with various contents of hybrid and single-phase nanoparticles. Then, the composite samples underwent tensile tests to thoroughly examine their tensile properties.

2. Material and Method

2.1 Materials

Nano silica particles from Graphene Chemical Industries Co., Turkey, and multiwalled carbon nanotubes from Nanografi Nanotechnology Co., Turkey, were utilized as a reinforcement phase. Epoxy matrix used in the production of nanocomposite in this study was purchased from Dost Chemical Co., Turkey. To create the thermosetting epoxy resin system, Momentive MGS L160 and an amino-acid-based hardener MGS H160 were utilized. The resin system was then mixed at a 100:35 mass mixing ratio. The physical properties of the materials used in the study are presented in Table 1 [26].

2.2 Preparation of the Nanocomposite Samples

The effect of MWCNT and NS on the mechanical behaviors of composite was systematically investigated by producing composites with different nanofiller configurations, both single and in hybrid combinations, at specific weight ratios. The study involved the incorporation of 0.1, 0.3, 0.5, and 0.7 wt.% MWCNT and, 0.5, 1.0, and 1.5 wt.% NS for single-filled configurations. In accordance with the findings from Rahmanian et al., it was identified that the presence of nanoparticles exceeding 1 wt.% led to a disturbance in the distribution morphology of combined fillers. Considering this observation, hybrid nano-filled (binary combination of MWCNT/NS) mixtures were carefully formulated at a concentration of precisely 1% by weight within the epoxy matrix. This strategic approach was undertaken to ensure optimal compatibility and nano filler distribution, thereby maintaining the integrity and performance of the epoxy matrix [27]. Thus, hybrid nano-filled configurations were produced with nanofiller additives at hybridization ratios of 1:1, 1:2, and 1:3 (MWCNT: NS). A total of eleven different mixture types of nanocomposite samples were produced. A naming convention detailed in Table 2 was used to determine the nanoparticle contents and weight ratios of the samples.

To improve the nanofiller dispersion and reduce the viscosity of the epoxy resin, 99% pure acetone was introduced to nanoparticle- epoxy mixture as an organic solvent. Previous studies in the literature have indicated that the inclusion of acetone does not trigger chemical alterations in the epoxy chain [13,22]. The mixture was then homogenized at 8000 rpm until the acetone had completely evaporated from the matrix. Once this was confirmed by weighing, a hardener was added to the mixture and homogenized again for 15 minutes at room temperature. After the mixtures were prepared, they were poured into silicone molds, and air bubbles in the mixtures were removed using a heat gun. The mixture was left in the mold to cure at room temperature for 24 hours. Lastly, the nanocomposite samples were post-cured for 4 hours at 60°C. The summary of production steps is illustrated in Figure 1.

2.3 Tensile Test Procedure

Tensile tests were performed at room temperature using the SHIMADZU AG-X series universal testing machine, as shown in Figure 2, following the guidelines of ASTM D638. Dog bone-shaped test samples were used in the test, and the crosshead speed was set to 2 mm/min. the findings, at least five samples were examined, and average values were compared with one another.

Table 1. The physical properties of MWCNT, NS particles and epoxy resin system [26].

Material	Specification	Values
MWCNT	Purity *(%)* Density *(g/cm^3)* Surface Area *(m^2/g)* Inner diameter *(nm)* Outer diameter (*nm*) Length *(µm)*	>96 2.4 >210 5-10 8-18 10-35
NS	Purity *(%)* Density *(g/cm^3)* Surface Area *(m^2/g)* Average particle size *(nm)*	99.5 0.05 300 15-35
MGS L160 Resin	Density *(g/cm^3)* Viscosity *(mPa.s)*	1.13 - 1.17 700 - 900
MGS H160 Hardener	Density *(g/cm^3)* Viscosity *(mPa.s)*	0.96 - 1.00 10 - 50

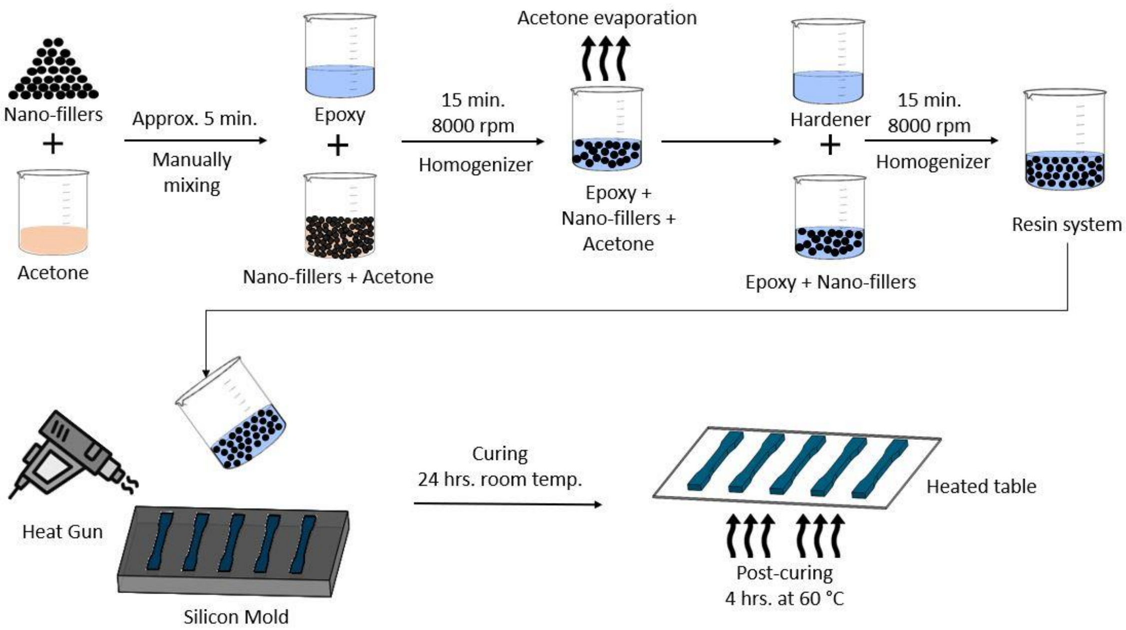

Figure 1. Nanocomposite production steps.

Table 2. Composition of nanocomposite samples

Nanoparticle Type	Designation	Filler Ratio (wt%)
-	Pure	0
MWCNT	C1	0.1
	C2	0.3
	C3	0.5
	C4	0.7
NS	S1	0.5
	S2	1.0
	S3	1.5
MWCNT : NS	H1	0.5 : 0.5
	H2	0.33 : 0.66
	H3	0.25 : 0.75

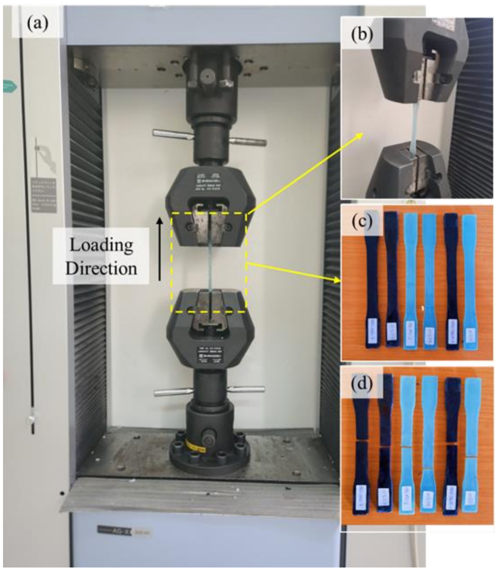

Figure 2. (a) Tensile test set-up, (b) boundary conditions, (c) test samples -C3, C4, S2, S3, H1 and Pure-, (d) fracture test samples.

3. Results and Discussion

Tensile testing at room temperature, in accordance with ASTM D638, was conducted on pure epoxy, as well as single and hybrid nanoparticle-filled epoxy composites. Figure 3 depicts the engineering stress-strain curves of both nanoparticle-filled and unfilled samples. The samples exhibit a linear trend up to the ultimate tensile strength before experiencing sudden drops, indicating catastrophic failure in the composite samples.

The average results of the tensile tests are presented in Table 3 and Figures 4a- b. Notably, an enhancement in tensile strength was observed across all samples in comparison to the pure epoxy sample. Specifically, when focusing on single nanoparticle-filled epoxy composites, the maximum improvements in tensile strength were found as 28.35% and 21.25% for the C3 and S2 samples containing 0.5 wt.% MWCN and 1 wt.% NS, respectively, as compared to the pure epoxy sample. In the context of other nanocomposite types, increases in tensile strength values were observed, measuring 3.77%, 11%, and 25.89% for C1, C2, and C4 samples, respectively, incorporating 0.1, 0.3, and 0.7 wt.% MWCNT. Slight enhancements were computed at 4.37% and 3.7% for S1 and S3 samples, respectively, including 0.5 and 1.5 wt.% NS. This may be due to the morphology of MWCNT in tube form and NS in spherical form. MWCNT forms an interconnected structure with a complex tube shape, resulting in greater load transfer between nanotubes and epoxy [28,29]. The noteworthy aspect of the data presented in Table 3 and Figure 4a is the consistent and substantial improvement observed in the tensile strength of all hybrid nanoparticle-filled samples.

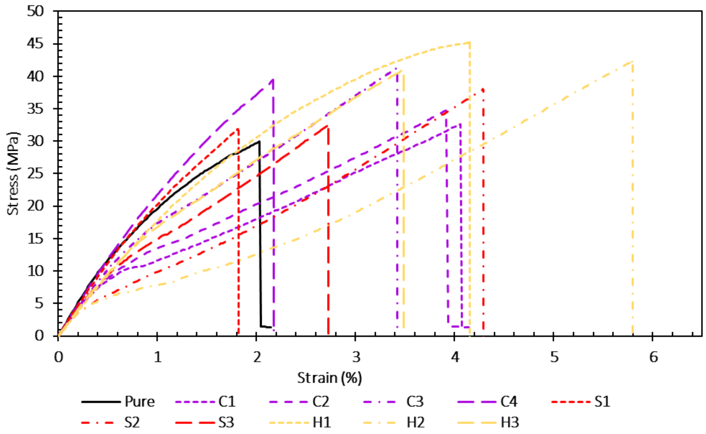

Figure 3. Engineering stress-strain curves of nanocomposites

Table 3. Improvements in tensile behavior of the nanocomposites

Sample	Tensile Strength (MPa)	Standard Deviation in Tensile Stress (MPa)	Tensile Strain (%)	Standard Deviation in Tensile Strain (%)	Tensile Modulus (GPa)
P	31.32	0.44	2.14	0.32	1.53
C1	32.50	1.95	3.90	1.37	1.40
C2	34.77	1.78	4.02	1.19	1.14
C3	40.20	0.75	3.46	0.24	1.16
C4	39.43	2.57	2.24	0.12	2.61
S1	32.69	2.03	1.45	0.20	2.70
S2	37.98	5.45	4.19	0.50	4.37
S3	32.50	1.99	2.80	1.07	1.30
H1	45.18	1.92	4.01	0.60	2.13
H2	42.57	2.25	5.20	1.27	2.27
H3	41.53	3.19	3.78	0.39	2.25

This observation strongly implies a positive hybrid effect, where the combination of different nanoparticle types leads to enhanced tensile properties compared to single nanoparticle-filled samples or the pure epoxy sample. As can be seen from the test results presented in Table 3, an excellent improvement in tensile strength was recorded as 44.26% in H1 hybrid sample with 1:1 nanoparticle ratio. Also, in H2 and H3 samples, these improvements were found as 35.91% and 32.60, respectively. The enhancement in tensile strength attributed to the introduction of nanofillers stems from the expanded interfacial region, thereby mitigating crack energy dissipation.

Also, the improved tensile strength observed in composites filled with either single or hybrid MWCNT and NS is a result of the better wettability of these nanofillers within the epoxy matrix. This improved wettability is a result of the effective and uniform distribution, and improved bonding strength established between the epoxy resin and the nanofillers [30,31]. This phenomenon significantly contributes to the overall enhancement in mechanical properties observed in the composite material. On the other hand, up to a certain nano-particle content, the tensile strength values begin to drop. The reason for this can be related to as the nanoparticle filler ratio is increased, the van der Waals attractive forces between the particles increase, thus resulting in very high surface energies, and the particles tend to aggregate to reduce this energy [26,32]. Thus, these agglomerations cause nonuniform local stress concentrations.

Strain plays an important role as a fundamental parameter, offering valuable insights into the ductility of materials. As presented in Table 3 and Figure 4b, tensile strain values were improved except for the S1 sample compared to the pure epoxy sample. Specifically, the addition of MWCNT and NS as single nanofillers resulted in substantial improvements of about 87.85% and 46.26% for the C2 and S2 samples containing 0.5 wt.% MWCNT and 1 wt.% NS, respectively, as compared to the pure epoxy sample. This can be attributed to the inhomogeneous dispersion and agglomeration of the nanofillers and their embedded into the epoxy. Examining the tensile strain values of hybrid nanofillers, H1, H2, and H3 showed impressive improvements of approximately 87%, 143%, and 76%, respectively. This underscores that the combined application of MWCNT and NS significantly enhances the ductility of the epoxy matrix, mitigating embrittlement.

3.1 Fracture Characterization

The fracture surfaces of the samples were examined to better understand the behavior of the samples subjected to tensile loads and to examine the fracture modes. The surfaces of the samples were imaged with high-resolution microscopes (LEICA EZ4 HD and LEICA DM750). Figures 5 and 6 represent the surface images of the test samples. In the presented figures, the left column images were taken at 35x magnification, while those in the right column were taken at 63x magnification.

When the surfaces of the unmodified samples are examined, the fracture surface is smooth and glassy, and no significant plastic deformation occurred during fracture. In Figure 5, the surfaces of nanoparticle-filled samples are rougher and more complex than pure samples. The greater the roughness, the more complex the fracture mechanisms and the more energy absorption during crack propagation, leading to an enhancement of the matrix's mechanical characteristics [26,29]. When the fracture surfaces are compared, the surface of MWCNT samples is rougher and thus the strength values of these samples are higher. This is due to the entangled tube form of MWCNT particles, which increases the load transfer by making more interfacial bonds with the matrix.

However, as the nanoparticle content exceeded a specific weight percentage (0.5 wt.% for MWCNT and 1 wt.% for NS), stress concentrations, in the form of star-shaped fracture patterns, indicating fracture origin sites on the surfaces, became evident. This ultimately led to the initiation of cracks. In the literature, these stress concentrations are often attributed to nanoparticle agglomeration [33,34].

As seen Figure 6 when the surfaces of the hybrid nanoparticle samples were examined, the surface of the H1 samples was rougher, and this roughness caused more energy absorption and showed better mechanical improvement than the other hybrid samples. This may be due to the morphological structure of MWCNT as mentioned above. It has been noted that large tensile stresses arise in matrix structures reinforced by high aspect ratio additions, like nanotubes because the filler merges with the matrix. High energy absorption and a rise in fracture energy were caused by the frictional forces attempting to keep the nanotubes from separating from the matrix and the shear force created by the separation zone expanding along the nanotube length [35].

As a result, the rougher the fracture surfaces, the more complex the fracture modes will be, resulting in higher energy absorption and higher energy fracture, increasing the mechanical properties.

4. Conclusions

This study investigated the influence of incorporating nanoparticles into the epoxy matrix on the load-carrying capacity of the material. The study assesses different weight contents of MWCNT and NS particles. Results demonstrate that the nanofillers incorporation into the matrix significantly impacts the load-carrying capacity. The results obtained are as follows.

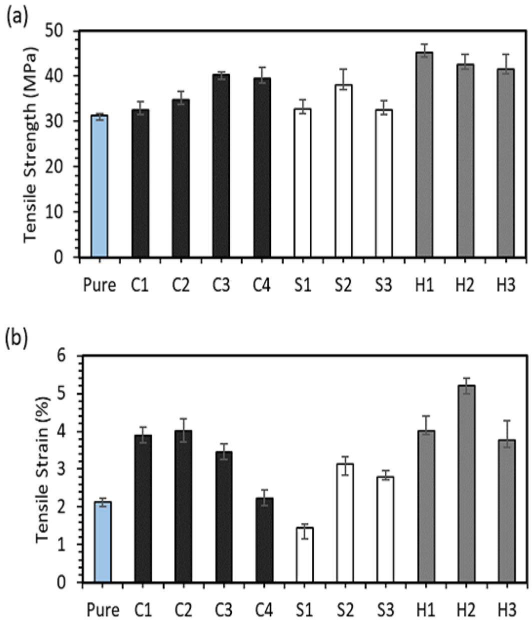

Figure 4. Tensile test results, (a) tensile strength, (b) tensile strain deviation for nanocomposites

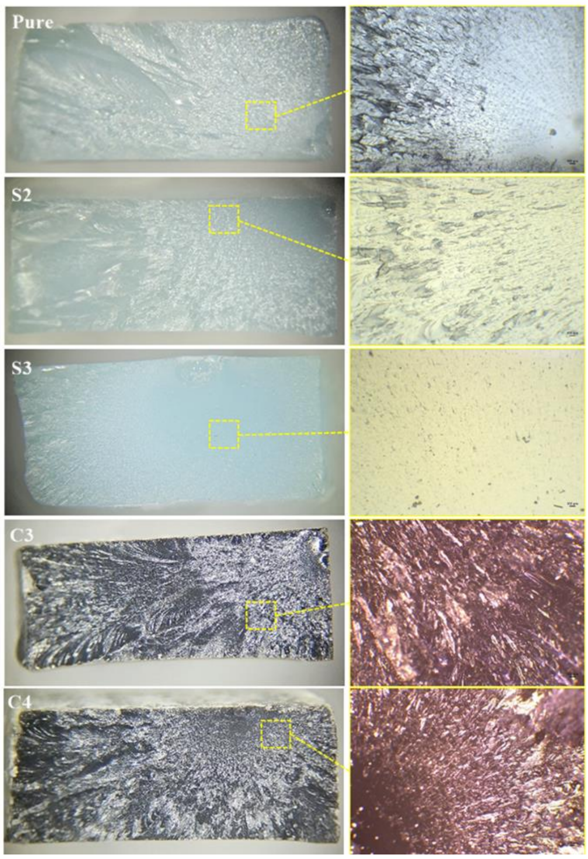

Figure 5. High-resolution microscope images of single nanoparticle filled samples

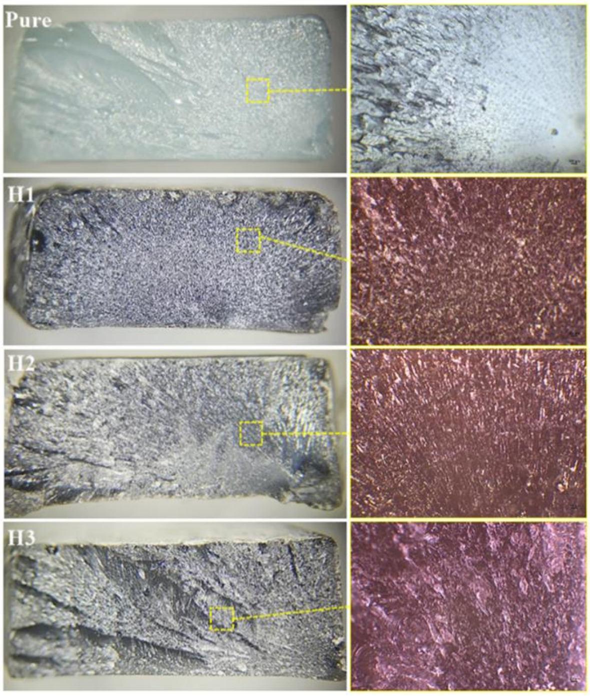

Figure 6. High-resolution microscope images of hybrid nanoparticle filled samples

- Tensile strength demonstrated improvements in all nanocomposites containing single nanoparticles. Among these nanocomposites, the maximum tensile strengths were 40.20 MPa and 37.98 MPa in C3 and S2 samples, respectively, and an improvement of 28.35% and 21.25% was achieved.
- Significant enhancements were observed in the tensile strength values of all composite samples that contained hybrid nanofillers. The H1 sample, which had a 1:1 hybrid ratio, showed the highest improvement in tensile strength value, with a 44.26% increase compared to the pure epoxy sample. The H2 and H3 samples also showed improvements in strength values, with increases of 35.91% and 32.60%, respectively.
- An improvement in the ductility of the material was observed in almost all nanocomposite samples, as indicated by the increase in tensile strain values. Maximum improvement in strain value was recorded as about 143% in H2 hybrid sample.
- From the test results, we can conclude that by opting for more cost-effective materials, we can produce composite materials with enhanced mechanical properties. Although the unit cost of NS is much less than that of MWCNT, it is possible to achieve superior mechanical properties in a composite material by using equal or higher amounts of NS in the form of a hybrid nanofiller.
- Further research could also be conducted to determine the effectiveness of using a hybrid nanofiller combined with a nanoparticle having tube shape morphology (CNT) and different types of morphology such as plate-like nanoparticles graphene nanoplatelets (GNP).

Declaration

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article. The author also declared that this article is original and was prepared in accordance with international publication and research ethics, and ethical committee permission or any special permission is not required.

Author Contributions

A. Kayalar and N.F. Dogan have contributed to defining and managing the conceptual and design processes of the study, data collection, data analysis and interpretation, drafting the article, critically examining the conceptual content, and contributing to the final approval and full responsibility stages.

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