




RESEARCH ARTICLE

THE MECHANICAL CHARACTERIZATION OF CARBON BASED NANOPARTICLE
REINFORCED EPOXY COMPOSITES: A COMPARATIVE STUDY

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Abstract

This comparative study experimentally investigates the effect of both the type and content of carbon based nanoparticles on the mechanical properties of epoxy composites. For this purpose, carbon nanotubes (CNTs), expanded graphite (EG), and carbon black (CB) were used as reinforcing nanoparticles at various concentrations within the epoxy polymer. The nanoparticles were dispersed by ultrasonication method. CNTs incorporated up to 0.4% by weight (wt.) while EG and CB nanoparticles were employed at 4%, 8%, 10%, and 12% concentrations by weight. Tensile tests of the nanocomposites were conducted according to ASTM D680 to determine the mechanical properties of nanocomposites including ultimate tensile strength and modulus. The results revealed that all types of nanoparticles have a strong reinforcing effect on the mechanical properties depending on their concentrations. When carbon nanotubes (CNTs) were used, the highest improvement in strength, by 84.7% at 0.1% wt., and in modulus, by 32.1% at 0.2% wt. content, was observed. EG nanoparticles exhibited improvement in both strength and modulus at all contents. The highest improvement in strength, by 109.6% at 4% wt., and in modulus, by 95.6% at 10% wt. concentration, was observed. In the case of carbon black (CB), improvement in strength was observed only at 4% wt. concentration, by 44.9%. On the other hand, enhancement in modulus was seen at all CB contents, with the greatest improvement at 10% wt., reaching 58.2%.

Keywords

Nanocomposites,
Mechanical properties,
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Expanded graphite,
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Time Scale of Article

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1. INTRODUCTION

Due to their high modulus, strength, electrical insulating properties, and chemical stability, epoxy resins have found extensive application in structural engineering as adhesive or matrix materials in the aerospace, marine, and automotive industries [1–3]. The widespread adoption of epoxy materials in engineering applications is hindered by their inherent brittleness, a drawback resulting from their highly cross-linked microstructure [4, 5]. Therefore, enhancing the mechanical properties of epoxy is crucial to obtain functional composite materials.

One of the effective ways is to improve the mechanical properties of epoxy composites is to introduce a secondary phase of carbon-based nano fillers such as carbon nanotube (CNT) [6], silicon carbide (SiC) [7] carbon black (CB) [8], expanded graphite (EG) and graphene nanoplatelet (GNP) [9] within epoxy matrix. Beside the improvement in the mechanical properties, the addition of nanoparticles into the polymer materials enhance the thermal [10] and electrical properties [11] of the epoxy phase as well. CNTs have gained widespread popularity due to their remarkable mechanical [12], electrical [13], and thermal properties [14]. CNTs are nano-sized cylindrical structures made of carbon. CNTs have gained widespread popularity due to their remarkable mechanical [12], electrical [13], and thermal properties [14]. CNTs are nano-sized cylindrical structures made of carbon. They can exist as either single-walled

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carbon nanotubes (SWCNTs), comprising a single graphene layer rolled into a cylindrical shape; double-walled carbon nanotubes (DWCNTs), composed of exactly two single-walled carbon nanotubes, one nested in another; or multi-walled carbon nanotubes (MWCNTs), consisting of multiple concentric cylindrical layers of graphene. They are extensively employed in CNT/polymer composites for both functional and structural applications [15, 16]. EG is a versatile carbon-based nanomaterial characterized by its low density, high aspect ratio, porous structure, excellent thermal and electrical conductivity, ease of preparation, and cost-effectiveness [17]. Consequently, numerous studies have explored the incorporation of EG as a functional filler in polymer composites for diverse applications, including conductive polymer components, high-temperature resistant parts, sealing gaskets, and flame-retardant composites [18–22]. CB produced by the incomplete combustion of carbonaceous or petroleum products stands out as one of the primary reinforcing fillers within the polymer industry due to its readily available source, lightweight nature, high electrical conductivity, and low cost [23, 24]. Possessing superior characteristics such as the ability to absorb UV energy and high electrical and thermal conductivity make CB an excellent candidate to form functional thermoset composites. CB essentially consists of elemental carbon in the form of extremely fine particles with a partially amorphous molecular structure. The typical size of CB particles ranges from 5 to 100 nm, however they have tendency to form cluster resulting in bigger size aggregates up to 500 nm.

The reinforcing effect of the carbon-based nanoparticles on the mechanical properties depends on some parameters such as morphological structure, particle size, distribution level, and volume fraction of nanoparticles in the epoxy structure [14]. CBs, CNTs, and EGs can be classified as 0D, 1D, and 2D based on their morphological structure, respectively. This diversity on the morphological structure might lead to the different surface areas in these nanoparticles. The surface area of nanotubes can act as desirable interface for efficient stress transfer from the epoxy matrix to the nanoparticles [25]. In other words, the higher the interfacial area between the nanoparticles and the matrix phase, the greater load transfer capability. Table 1 lists some of the published works related to the mechanical performance of CNT, CB, and EG filled epoxy composites at various nanoparticle concentrations.

Table1. Some published works related to mechanical performance of CNT, CB, and EG filled epoxy composites.

Nanoparticle	Nanoparticle content (wt.%)	Mechanical property	Improvement (%)	Reference
DWCNT	1	Tensile modulus	6.4	[26]
SWCNT	0.3	Tensile strength	8.2	[27]
		Tensile modulus	5.5	
MWCNT	0.3	Tensile strength	-	[27]
		Tensile modulus	8.5	
SWCNT	1	Tensile strength	30	[28]
MWCNT	2	Tensile strength	24	[29]
		Tensile modulus	-	
MWCNT	3	Tensile strength	-	[30]
		Tensile modulus	16	
MWCNT	0.5	Flexural strength	29.5	[31]
		Flexural modulus	32.3	
CB	5	Flexural modulus	23	[32]
CB	2	Tensile strength	32	[13]
		Flexural strength	88	
		Impact strength	10	
CB	0.3	Flexural strength	8	[33]
			12	
CB	5	Flexural strength	22	[34]
CB	1	Tensile strength	4	[35]
		Tensile modulus	1	
EG	1	Tensile modulus	4	[18]
EG	9	Tensile strength	30	[36]
EG	2.5	Tensile strength	21	[37]
			12	
EG	2.5	Tensile modulus	10	[37]
			25	
EG	1	Impact strength	21	[38]

It is evident from the literature that, in general, CNTs have a reinforcing effect on the mechanical properties of epoxy resin at lower contents, up to 1% wt. Conversely, when using expanded graphite (EG) and carbon black (CB) as fillers, the enhancement in mechanical performance occurs at higher contents, beyond 1% wt. Additionally, while the electrical percolation of CNTs ranges between 0.1% and 0.5% [39], it falls within the range of 1% wt. and 10% wt. in CB/epoxy composites [40] and 1% wt. and 5% wt. in EG/epoxy composites [36]. This difference can be attributed to the high aspect ratio and intrinsically high electrical conductivity of CNTs, resulting in significant improvements in both the mechanical and electrical properties of polymer composites at low contents. Conversely, the comparatively lower surface area of CB and EG nanoparticles requires higher contents for efficient functionality in polymer composites. Therefore, understanding the effect of various nanoparticles, such as MWCNTs, CBs, and EGs, with different morphological structures on the mechanical properties of epoxy nanocomposites at various nanoparticle contents is crucial. However, the effect of nanoparticle contents on the mechanical properties of these epoxy nanocomposites has not been comparatively studied to the best of the author's knowledge. Hence, this study aimed to conduct comparative experimental work to investigate the effect of nanoparticle concentration on the tensile strength and elasticity modulus of MWCNT, CB, and EG reinforced epoxy composites. For this purpose, epoxy composites filled with 0.1% wt., 0.2% wt., 0.3% wt., and 0.4% wt. MWCNTs, 4% wt., 8% wt., 10% wt., and 12% wt. CBs, and 4% wt., 8% wt., 10% wt., and 12% wt. EGs were manufactured. The ultrasonication method was employed for nanoparticle distribution within the epoxy. There are two main methods for the distribution characterization of nanoparticles: these are direct microscopic observation [41] and indirect estimation methods [42]. For a good nanoparticle distribution characterization, it is necessary to use direct observation and indirect estimation methods together. In the direct microscopic method, observations are made using methods such as optical microscope (OM), scanning electron microscope (SEM), atomic force microscope (AFM), and transmission electron microscope (TEM). On the other hand, the electrical resistance measurement technique is one of the indirect estimation methods of nanoparticle distribution quality within polymer materials [43]. In this study, the dispersion quality of nanoparticles within epoxy resin was measured based on the electrical resistance measurement. Subsequently, tensile tests were performed to evaluate the mechanical properties of these nanocomposites.

2. MATERIALS AND METHODS

The epoxy-based matrix material used in this study was sourced from Duratek™, Turkey. This matrix consisted of two components: DTE 1000 resin and DTS 1105 hardener. The weight ratio for mixing DTE 1000 resin to DTS 1105 hardener was 100:35. Multi-wall carbon nanotube (MWCNT), expanded graphite (EG), and carbon black (CB), were employed as carbon nanofillers in the epoxy composites. MWCNT nanoparticles were supplied from Nanografi Co., Ltd., Turkey. EG (TIMREX® C-THERM™301) and CB (ENSACO) were kindly provided by TIMCAL (Switzerland). Table 2 presents selected physical properties of the fillers.

Table 2. The physical properties of the nano fillers.

Property	CNT	CB	EG
Density (g/cm ³)	0.25	0.182	0.19
Surface area (m ² /g)	250-300	66.2	27
Particle size	diameter: 8-10 nm length: 1.5 μm		

2.1. Dispersion Process

Nanoparticle dispersion within a polymer matrix has been accomplished using several methods, including ultrasonication, calendaring, and high shear mixing [13]. Among these techniques, ultrasonication is a commonly utilized and effective method for dispersing carbon based nanoparticles

in polymers [44]. Since epoxy has a high viscosity, its viscosity is required to be reduced using a solvent such as acetone for efficient dispersion of nanoparticles. In this study, no solvent was employed in the dispersion process of nanoparticles through ultrasonication to eliminate the residual solvent effect on the mechanical performance of nanocomposites for the purpose of obtaining reliable test data. Therefore, strong ultrasonication process was performed at 150 W for 20 min to disperse the nanoparticles in small amount of epoxy of 50 g as seen in Figure 1.



Figure 1. Ultrasonication process

2.2. Optical Microscopy

The dispersion of nanoparticles within epoxy was characterized by optical images taken in transmission mode using a microscope (Nikon Eclipse) equipped with a camera having a 20X objective lens. Samples for optical imaging were prepared by pressing a small droplet of the composite mixture between two glass slides.

2.3. Electrical Resistance Measurement

Since the dispersion quality of nanoparticles within a polymer is strongly dependent on both sonication power and time, we selected the optimum ultrasonication power and dispersion time based on the indirect measurement of dispersion quality. This was achieved by measuring the electrical resistance of 0.2 %wt. MWCNT-filled epoxy mixtures that were dispersed at various ultrasonication powers and times. The electrical percolation threshold of MWCNT was preliminary found as 0.1 %wt. based on electrical conductivity measurements [6]. Therefore, 0.2 %wt. MWCNT was employed in the optimization process of ultrasonication. It is important to note that the ultrasonication parameters may vary for carbon black (CB) and expanded graphite (EG) nanoparticles due to their inherently different electrical conductivity and morphological dimensions. However, since multi-walled carbon nanotubes (MWCNTs) exhibit electrical conductivity at lower contents compared to CB and EG nanoparticles, an optimization study of ultrasonication parameters based on electrical resistance measurement was conducted using MWCNTs at a content of 0.2% by weight within epoxy. Rectangular specimens of about 70 mm in length, 10 mm in width, and 3 mm in thickness were molded for electrical resistance measurement as seen in Fig. 2. Whiskered carbon fibers were used at both ends of the specimens to obtain good contact with the filler network in the specimen. Electrical resistance measurement was achieved using a LCR meter. Two batches with three specimens were prepared for consistency. The measured resistance values are the functions of the geometrical aspects of the specimens such as length and the cross sectional area. Therefore, the measured resistance values were normalized to determine the specific electrical resistivity of the specimens using using equation 1. In this equation, ρ , R, A, and L represent sthe specific resistivity, measured resistance, cross – sectional area, and length, respectively.

$$\rho = \frac{RA}{L} \quad (1)$$

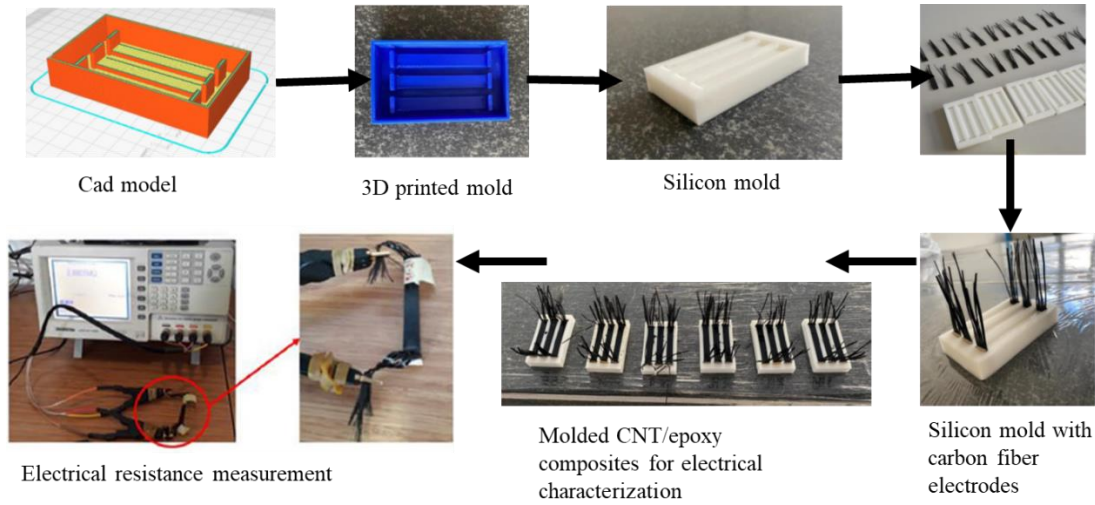


Figure 2. Manufacturing steps of specimens for electrical resistance measurement.

2.4. Tensile Tests

The tensile tests were carried out to investigate the effect of the nanoparticle content on the mechanical properties of nanoparticle reinforced epoxy composites. The rectangular tensile test specimens with the dimensions of $127 \times 12.7 \times 3.2$ mm were molded according to ASTM D680 as shown in Fig 3. Tensile tests were conducted using universal tensile testing machine at a displacement rate of 2 mm/min up to the specimen failure. The tensile tests were repeated three times for consistency. Displacements (ΔL) and loads (F) were automatically recorded using a computer system and consequently the stress (σ) and strain (ε) values were calculated using equation 2 and equation 3, respectively.

$$\sigma = \frac{F}{A} \quad (2)$$

$$\varepsilon = \frac{\Delta L}{L_0} \quad (3)$$

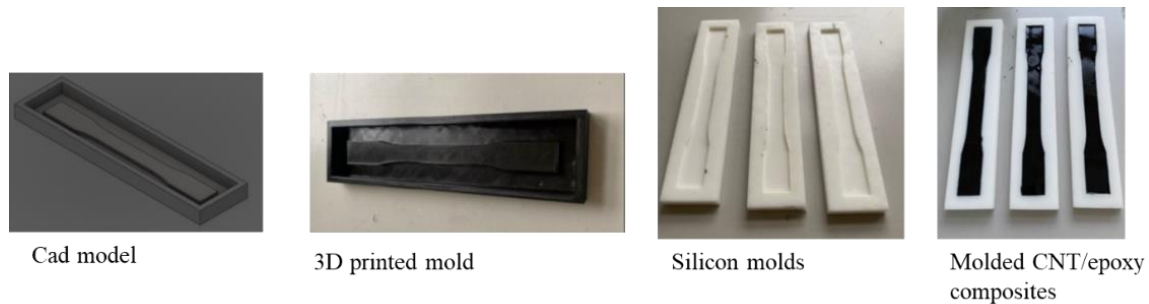


Figure 3. Manufacturing of tensile test specimens.

Table 3 lists the nanoparticle contents by weight for different nanoparticles used within the epoxy. Because CNTs possess a larger surface area compared to EG and CB nanoparticles, they exhibit stronger intermolecular interactions through van der Waals forces. This strong interaction increases the possibility of agglomerate formation within the CNTs and consequently results in a more challenging dispersion process due to the increase in viscosity. Therefore, CNTs were not utilized at concentrations higher than 0.4% by weight within the epoxy. Since EG and CB nanoparticles exhibit very low reinforcing effect at contents up to 0.5 % wt., the investigation aimed to explore the effect of nanoparticle content on mechanical properties at higher concentrations, particularly above the percolation threshold for these nanoparticles.

Table 3. The nanoparticle contents by weight for different nanoparticles used within the epoxy.

Nanofiller	Content (% wt.)
MWCNT	0.1-0.2-0.3-0.4
CB	4-8-10-12
EG	4-8-10-12

3. RESULTS and DISCUSSION

3.1. Optical Microscopy

Figure 4 shows the optical microscopy images of EG and CB filled epoxy blends at the concentrations of 4% wt. and 10% by weight. It is seen from the optical images at 4% concentration that EG-filled epoxy sample exhibits transparency, owing to its tendency to spread easily between glass plates compared to the CB-filled sample. This result indicates the fact that EG nanoparticles having lower surface area are less likely to increase the viscosity of epoxy compared to CB nanoparticles at the same concentration. The resulting high viscosity leads to challenges during ultrasonication process, causing nanoparticle agglomeration within epoxy mixture. Moreover, the formation of agglomerates is more likely to occur as the nanoparticle content increases. The optical images give some useful insight in macro scale about the effect of nanoparticle morphology on the dispersion quality. However, it should be noted that the dispersion quality of nanoparticle filled polymer resins needs to be examined in micro scale using SEM for better understanding of size and distribution of both nanoparticles and agglomerations within resin.

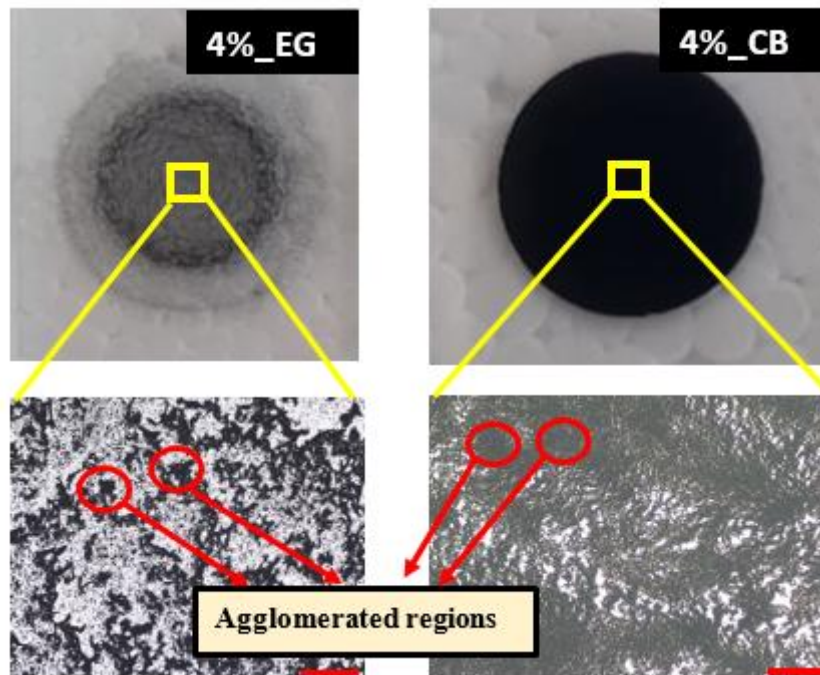


Figure 4. The optical images of CB and EG filled epoxy mixtures at 4% and 10% concentrations by weight. Scale: 32 μ m.

3.2. Electrical Measurement

The purpose of the electrical resistance measurement was to determine the optimum dispersion parameters of ultrasonication method. Figure 5 compares the specific electrical resistance values of 0.2

% wt. MWCNT-filled epoxy composites dispersed at varying power for 20 min as a column bar. The average values of electrical resistance change were determined from two batches of epoxy composites filled with 0.2% wt. MWCNTs, utilizing a total of six specimens.

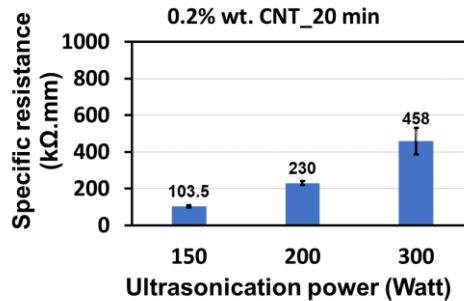


Figure 5. The specific electrical resistance values of 0.2 %wt. MWCNT-filled epoxy composites dispersed at varying power for 20 min.

The results show that the lowest and highest resistance is seen at 150 W and 300 W, respectively. Since it is known that the better dispersion of MWCNTs in epoxy results in lower electrical resistivity (or higher conductivity) the ultrasonic dispersion at a greater power than 150 W is not efficient in terms of dispersion quality. Therefore, the effect of the dispersion time was investigated on the dispersion quality in terms of electrical resistivity at 150 W. Figure 6 compares the specific electrical resistance values of 0.2 %wt. CNT-filled epoxy composites dispersed at 150 W power for varying dispersion time as a list and column bar. The results indicate that the lowest resistance is seen at 20 min of dispersion time. Based on these findings, the optimum dispersion parameters of nanoparticles in epoxy matrix was decided as 150 W for power and 20 min for dispersion time. It would be expected that the higher ultrasonication power and the dispersion time, the greater the electrical conductivity and consequently better dispersion quality. However, the electrical properties do not only depend on the dispersion level but also the aspect ratio of the filler material. Therefore, these results in this study might be due to reduction in the aspect ratio of CNTs as a result of aggressive dispersion process of ultrasonication at higher ultrasonication power and longer dispersion time as discussed in the literature [45].

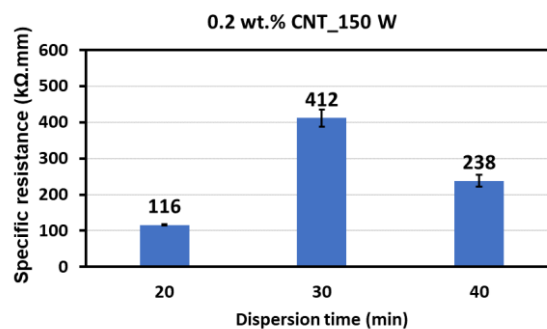


Figure 6. The specific electrical resistance values of 0.2 %wt. CNT-filled epoxy composites dispersed at 150 W for varying dispersion time.

3.3. Tensile Tests

The column graphs comparing the average mechanical properties for different CNT contents are seen in Figure 7. The results indicate that the incorporation of CNTs into the epoxy results in enhancement in the mechanical properties of the epoxy material. The improvement in the mechanical strength was

achieved as 84.7%, 62.9%, 25.1%, and 4.2% for 0.1%, 0.2%, 0.3%, and 0.4% CNT content by weight, respectively. In addition, the improvement in the elasticity modulus was realized as 32.1%, 9%, and 11.4% for 0.1%, 0.2%, and 0.3% CNT content by weight, respectively. On the other hand, the slight reduction in the elasticity modulus was observed in the case of using % 0.4 CNT content. The results indicate that the reinforcing effect on the mechanical properties of the epoxy decreases with increasing CNT content. CNTs are one dimensional nanoparticles with a very high aspect ratio (length/diameter) leading strong van der Waals forces between individual nanotubes. Therefore, CNTs tend to agglomerate due to this strong van der Waals forces [46]. This tendency needs to be overcome by dispersion techniques such as ultrasonication to obtain mechanically strong and electrically and thermally conductive functional materials for practical applications. However, this task is challenging, particularly at higher CNT contents due to the significant increase in the viscosity resulting in poor dispersion quality of CNTs within the epoxy. Hence, the larger agglomerates at the higher CNT contents lead to reduction in the load bearing capacity acting as defects within the structure.

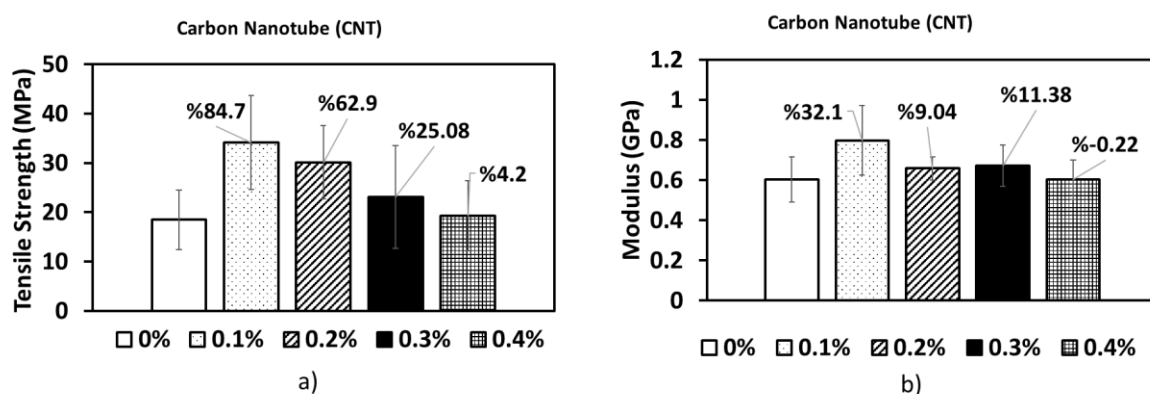


Figure 7. a) The average a) tensile strength and b) elastic modulus for different CNT contents.

Figures 8 and 9 compare the effect of nanoparticle type on the tensile strength and elasticity modulus for EG and CB nanoparticles at various concentrations. Since the CNTs were not employed at concentrations higher than 0.4 wt.%, only the EG and CB nanoparticles are included in these graphs. It is seen that the reinforcing effect of the EG on both the strength and modulus is significantly greater than that of CB at all concentrations. This might be attributed to the fact that the 2D nature of EG nanoparticles, providing a higher aspect ratio, results in a greater reinforcing effect compared to CB nanoparticles, which have a lower aspect ratio due to their spherical dimension. The results indicate that the incorporation of EGs into the epoxy results in enhancement in the mechanical strength as 109.6%, 87.4%, 73.3%, and 58.2% for 4%, 8%, 10%, and 12% EG content by weight, respectively. In addition, the improvement in the elasticity modulus was realized as 3.3%, 59.3%, 95.7%, and 65.1% for 4%, 8%, 10%, and 12% EG content by weight, respectively. The reinforcement effect of EG is seen at higher EG contents as opposed to CNTs. This is due to the fact that EG has lower aspect ratio and surface area compared to CNTs leading to better dispersion of EG nanoparticles without significant increase in the viscosity at higher concentrations. The strong reinforcing effect of EG is the clear indication of efficient mixing of the EG particles resulting in good filler/matrix adhesion [47]. Thanks to having very high modulus of the rigid graphite layers, significant enhancement in the modulus occurs as a result of EG modification in the epoxy [48]. Similar to the CNT modified composites, the reinforcing effect seems to reduce at the higher EG content due to the fact that viscosity at the higher EG content prevents the better dispersion of individual EG particles within the epoxy.

The results show that the improvement in the strength is observed at %4 CB content by %45 indicating that the CB is a promising reinforcing nanoparticle. However, the higher CB content incorporation into the epoxy leads to reduction in the epoxy strength. This reduction in the strength at the higher CB content might be attributed to viscosity increase at higher CB contents [49] which also are the case for the CNT

and EG nanoparticles. CBs have a high tendency to form large aggregates due to the strong Van der Waals force at higher concentrations. Large agglomerates cause bad interfacial interaction between epoxy phase and decrease the mechanical properties. Moreover, the high viscosity at high CB contents prevents the easy movement of CB particles and causes the non-uniform distribution of CB particles within epoxy. On the other hand, the improvement in the modulus was observed at all CB contents. This improvement in the elasticity modulus was realized as 55.3%, 13.6%, 58.2%, and 36.9% for 4%, 8%, 10%, and 12% CB content by weight, respectively. It should be noted that there is no correlation between the CB content and the enhancement level in the modulus.

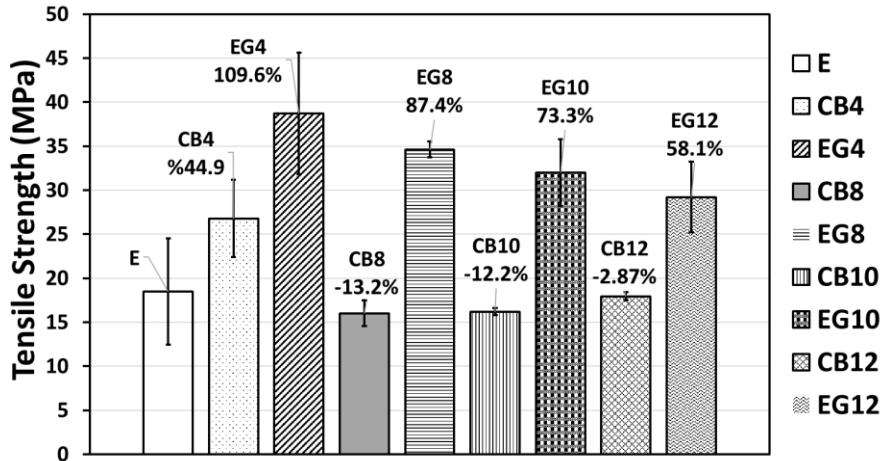


Figure 8. The average tensile strength of nanoparticle filled epoxy composites.

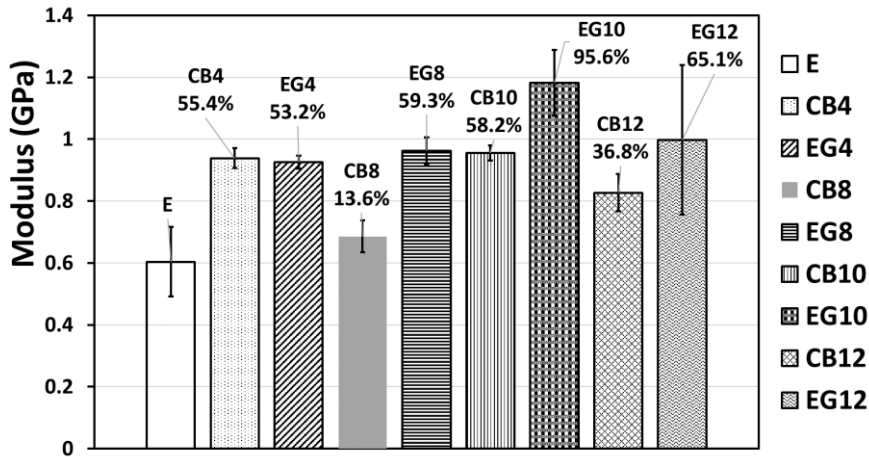


Figure 9. The average tensile modulus of nanoparticle filled epoxy composites.

4. CONCLUSION

In this study, a comparative experimental study was conducted to investigate the effect of the nanoparticle type and content on the mechanical properties of the epoxy composites. The main conclusions of this study were summarized as follows:

In general, all type of nanoparticles exhibit improvement on the mechanical properties of the epoxy composites at different concentrations.

CNTs were used up to 0.4% by weight due to the challenges in achieving proper dispersion at higher concentrations. The improvements in the strength were observed as 84.7%, 62.9%, 25%, and 4.2% for 0.1 % wt., 0.2 % wt., 0.3 % wt., and 0.4 % wt. CNT concentrations, respectively. The enhancement in the modulus was achieved as 32.1%, 9%, and 11.4% for 0.1 % wt., 0.2 % wt., and 0.3 % wt. CNT concentrations whereas an impairment in the modulus was realized at 0.4% wt. CNT concentration.

The improvements in both the strength and modulus were observed at all EG concentrations. This improvement in the strength was achieved as 109.6%, 87.4%, 73.3%, and 58.2% for 4 % wt., 8 % wt., 10 % wt., and 12 % wt., EG concentrations, respectively. Similarly, the modulus showed enhancements of as 53.2%, 59.3%, 95.6%, and 65.1% for the same concentrations.

Unlike the EG, the reinforcing effect of CB nanoparticles on the strength was seen only at 4 CB % wt . concentration by 44.9%. The reduction in the strength was observed by 13.2%, 12.2%, and 2.9% for 8 % wt., 10 % wt., and 12 % wt. CB concentrations, respectively. On the other hand, the improvement in the modulus at all CB concentrations was achieved as 55.4%, 13.6%, 58.2%, and 36.9%, for 4 % wt., 8 % wt., 10 % wt., and 12 % wt., CB concentrations respectively.

The highest improvement in the strength was realized as 84.7%, 109.6%, and 44.9% for 0.1 % wt. CNT, 4 % wt. EG, and 4% wt. CB nanoparticles, respectively. The improvement in modulus occurred as 32.1%, 95.6%, and 58.2% for 0.1% wt. CNT, 10% wt. EG, and 10% wt. CB contents, respectively. On the other hand, the least reinforcing effect on the strength was found as 4.2% and 58.2% at 0.4% wt. and 12% wt. contents for CNT and EG nanoparticles, respectively. No enhancement was observed in the strength for CB nanoparticle beyond 4% wt. content. The tendency of exhibiting a weaker reinforcing effect at higher nanoparticle concentrations can be attributed to the higher viscosity at these concentrations, which hinders efficient nanoparticle dispersion, promotes agglomeration, and ultimately leads to a reduction in mechanical properties.

The reduction of reinforcing effect on modulus was not found proportional with increasing nanoparticle content which is case in the strength. This might be due to the fact that very high stiffness of nanoparticles increases the resistance against deformation.

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CONFLICT OF INTEREST

The author stated that there are no conflicts of interest regarding the publication of this article.

CRedit AUTHOR STATEMENT

Fatih Turan: Formal analysis, Writing - original draft, Visualization, Conceptualization, Investigation, Writing – Original Draft, Funding acquisition, Methodology.

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