

Influence of CNT Particle on Mechanical Properties of Epoxy Composites

T. Malyadri^{1,2}, J. Suresh Kumar¹ and M. Nagamadhu^{3*}

¹Department of Mechanical Engineering, Jawaharlal Nehru Technological University, Hyderabad - 500085, Telangana, India

²Department of Mechanical Engineering, VNRVJIET, Bachupally, Hyderabad, Telangana, India

³Department of Mechanical Engineering, BMS Institute of Technology and Management, Bangalore - 560064, India; nagamadhu74@gmail.com

Abstract

In the recent years very much rapid development in the field of nanotechnology in many applications like structural, medical, biotechnology, agriculture etc. Polymers and polymer based composites attain a vital role in structural applications due to its light weight, easy of handling etc. However, during processing of polymer few problems associated with crosslinking of polymer bonds. These problems are overtaking by reinforcing Nano particles to the polymers. This research work focusses on studying the mechanical properties of epoxy polymer composites by reinforcing various weight percentage of Carbon Nanotube (CNT). The epoxy composite is prepared by magnetic stirring, mechanical stirring using hand layup method followed by post curing. The one-way ANOVA used to study the descriptive statistics influence of CNT reinforcement from 0.2 to 0.8 weight percentage in a 0.2 intervals. Further, Scheffe multiple comparison, Turkey HSD and Holm and Bonferroni multiple comparison tests are used to study the significant effect between the reinforcing percentage. It was observed that adding CNT to the composites improves the tensile strength and it is also observed from *F* and *P* values that reinforcement statically significant.

Keywords: Carbon Nanotube, Epoxy Composites, Magnetic Stirring, Mechanical Property, Mines

1.0 Introduction

Nanoparticles have a wide range of applications, including biological and pharmaceutical delivery, gene therapy, tissue engineering, nanoscale biochips, DNA probes, microsurgical techniques, separation and purification of biomolecules, and cross-linking of polymer structures. Among these applications reinforcing in to structural parts having attractive applications due to better properties by adding nanoparticles. For the light weight composites epoxy polymer plays a significant role due to many advantages. Epoxies are less affected by water and heat; they also have high corrosion resistance than

other polymeric matrices. Epoxies include metal coatings, composites, uses in electronics, electrical components (such as chips on boards), LEDs, high voltage electrical insulators, brush manufacturing, fiber reinforced plastic materials, structural adhesives, and other uses. Sreehari *et al.* observed that epoxy having many applications due to its compatibility and crosslinking with other copolymers¹. Abhinay *et al.* worked on basalt natural fiber and marble fillers reinforcing polymer composites, and observed that tensile, flexural/bending and impact properties are improved by adding waste marble powders up to 7.5 weight percentage. Also observed that hardness and wear properties were enhanced by adding waste

*Author for correspondence

marble powders². Blandine *et al.* worked on physical aging of epoxy composites and suggested for many civil applications³. Ankit Rathi *et al.* worked on multiwalled carbon nanotubes (MWCNTs) to study the influence of thermomechanical properties and anticorrosive properties of epoxy composites. Observed that lap shear strength, tensile strength was improved by adding nanoparticle compared to neat epoxy. The anti-corrosion properties of mildsteel varnished with hybrid epoxy nanocomposites i.e ZrO₂/MWCNT (MNC) increases significantly with 1.0 wt% of 1.0 ZrO₂/MWCNT hybrid nanocomposites, with rate of corrosion increases upto 56×10^{-3} MPY from 8.82MPY⁴. CRN were added to the resin in the ratio of 0 wt%, 0.25 wt%, 0.5 wt%, 0.75 wt%, and 1 wt%. Composites were fabricated using vacuum-assisted resin perfusion (VARI) technology. The results showed an 18.35% increase in tensile strength when CRN was 0.5% by weight. The short-term strength was the highest, increasing by 38.96% at 0.25 wt%. The disk had the highest peak load, lowest absorbed energy, and highest stiffness when the CRN was at 0.5 wt%⁵. Sotirios *et al.* Various carbon nanoinclusions (soot, MWCNTs, graphene nanoplates, nanodiamonds) and different nanocomposites consisting of magnetite nanoparticles embedded in commercial epoxy resins with varying filler types and concentrations were addressed. Experimental data from DMA (Dynamic Mechanical Analysis) and static tensile tests shows that the storage modulus and tensile modulus storage modulus of the hybrid nanocomposite are unique to the filler properties and the epoxy matrix and nano-inclusions. The results show that the tensile strength and elongation at break of nanocomposites decrease with increasing filler content⁶. Farzaneh *et al.* Worked on multi-walled CNTs and found that incorporating this 0.15 wt% of the processed nanoparticles into the epoxy matrix significantly improved their thermo-mechanical properties when worked on multi-walled CNTs. It is moreover important, observing that there is an increase in storage modulus around 7.5% and 1.1%. Also the tensile strength is increased by about 70% and 40%, and the T_g decreased by about 12 °C - 16 °C. O-MWCNT-ZIF-8-H/EP or -8-CO/EP samples compared to unmodified epoxy samples (neat EP)⁷. Fati *et al.* Investigating the effect of CNTs on epoxy resin, the electrical conductivity of the non-conductive carbon fiber/epoxy sheet increases with

increasing additive ratio, as expected, reaching 120 S/cm for 1.25% CNT reinforcement. was found⁸. To analyze the properties of MWCNTs was blended into the resin at 0 (sample neat), 0.5, 1, 1.5, and 2 wt% by using the methods like, Rittin *et al.* reported mechanical, tribological (using a pin-on-disk tribometer), and morphological [Scanning and Transmission Electron Microscopy (SEM) of MWCNT-epoxy composites. TEM],. 0.5, 1, 1.5, and 2 wt%. The enhanced properties are shown in the results with 0.5 wt% of MWCNT-epoxy composites, but there are adverse effects on the properties when further increasing the MWCNT concentration⁹. Amir *et al.* worked on epoxy and CNT composites and observed that bonding strength of composite improved by adding CNT¹⁰. The vacuum bagging process improves the tensile, impact and flexural properties of hybrid composite laminates¹⁷. However, by adding nanoparticles high degrees of strength and stiffness, which registered with far less high-density material. When compared with neat epoxy, its barrier properties are better. Many of the researchers observed that by adding nanoparticles, manifest better properties than those micro-sized reinforcements. It is also observed that nano-reinforcements can exceptionally increase in the mechanical strength by effectively assisting particle hardening mechanisms. Mining companies can significantly improve the durability and strength of their equipment, leading to increased operational efficiency and cost savings. The extreme conditions i.e resist wear and tear, and ultimately increase productivity and efficiency in mining operations. By incorporating Composite technology into mining equipment and infrastructure, we enable unprecedented strength, durability, and reliability.

2.0 Material and Methods

2.1 Materials

An unfilled, unmodified epoxy resin - ARALDITE LY 556 is having pot life of upto 1 hour i.e. at 20°C and low viscosity i.e. 5000 - 8000 MPa at 20°C is mixed with hardener HY 951 of ratio 10:1. A conventional stirrer¹¹⁻¹⁴ for producing stirred composites with a setting of 50 rev/min. The Zenith Industrial Suppliers, which is located in Bangalore, India, had supplied resin and hardener named Araldite LY 556 and HY 951.

2.2 Manufacturing of Composites

By using compression conventional moulding technique, the reinforced epoxy composites are prepared. In the fabrication of each laminate of corresponding weight% of CNTs with fabric and resin mixture with hardener (as corresponding weight%) are used¹⁸. The polymer composites are found to be tough by varying their composition of CNT²⁰. The reinforced epoxy named Woven Sisal Fabric is of 0.2 to 0.8 weight percentages at 0.2 intervals. The nanoparticles mixed in acetone solution and then magnetic stirred, then poured in to epoxy and then stirred to get uniform mixing of nanoparticles. The mixture formed by the combination of resin and hardener in the good proportion¹⁹. To get better curing and post-curing, the hardener is mixed at ratio of 10:1. This is done at 80°C.

2.3 Mechanical Properties

According to ASTM D638-03, the tensile properties of composite samples are measured. It is measured by using the given size dimensions of 16 cm length, 1.25 cm width, and 0.4 mm thickness. At JSS University of Science and Technology, Mysore, India, the tensile test is performed at a speed of 1 cm/min with a gauge length of 10 cm. This process is done by using a universal testing machine.

2.4 Analysis Method

Differences between enhancement percentages are very difficult to examine and become significant when only 5 identical trials are used for each variation of

nanoparticles. Therefore, static methods are used to determine critical levels and other measures. Tukey's method used in combination with ANOVA is used to find means that are significantly different from each nanoparticle enhancement (group)¹⁵⁻¹⁶. Tukey's Honest Significance Test, or Tukey's HSD (Honest Significant Difference) method, is a one-step statistical technique and multiple comparison procedure. This technique is used to find means, which are remarkably different from each percent gain. The Scheffe method is a one-level multiple comparison procedure. The Scheffe method is applied to a set of estimates of all possible contrasts. These contrasts are between factor level means, which are not only just the pairwise differences considered by the Tukey-Kramer method. To address the issue of multiple comparisons the Holm-Bonferroni method is used. This method is also known as the Holm method or Bonferroni-Holm method. It is designed to control the FWER (Family-Wise Error Rate). This method is consistently more powerful than the Bonferroni correction.

3.0 Results and Discussion

3.1 ANOVA Analysis

The mechanical properties measured as per the standard mentioned in 2c, and maximum stress of each trial recorded in Table 1. The average, variance, standard deviations are mentioned in Table 2. The neat epoxy having tensile strength of 18.73 MPa, by adding CNT the percentage of stress enhanced is also shown. By adding 0.8

Table 1. Stress values of five identical trials are grouped in five groups to study the influence of CNT on epoxy.

CNT weight % (Five independent treatment)	Weight % of CNT				
	Neat epoxy 0%	0.2%	0.4%	0.6%	0.8%
Representation	(A)	(B)	(C)	(D)	(E)
Trial 1	18.390	20.364	25.654	32.542	39.684
Trial 2	19.452	21.652	26.354	33.254	40.653
Trial 3	17.487	20.324	25.684	32.654	41.657
Trial 4	17.654	19.985	27.654	33.587	40.654
Trial 5	20.654	22.356	24.668	32.986	38.345

Table 2. Descriptive statistics of your k=5 independent treatments

Treatment →	A	B	C	D	E	Pooled Total
Observations N	5.0	5.0	5.0	5.0	5.0	25.0
Sum $\sum x_i \sum x_i$	93.6390	104.6838	130.0166	165.0248	200.9951	694.3594
Mean \bar{x}	18.7278	20.9368	26.0033	33.0050	40.1990	27.7744
Sum of squares $\sum x_i^2 \sum x_i^2$	1,760.6951	2,195.8751	3,385.7164	5,447.3756	8,086.0491	20,875.7113
Sample variance s^2	1.7603	1.0338	1.2133	0.1847	1.5608	66.2631
Sample std. dev. s	1.3268	1.0168	1.1015	0.4297	1.2493	8.1402
Std. dev. of mean $SE_{\bar{x}}$	0.5933	0.4547	0.4926	0.1922	0.5587	1.6280

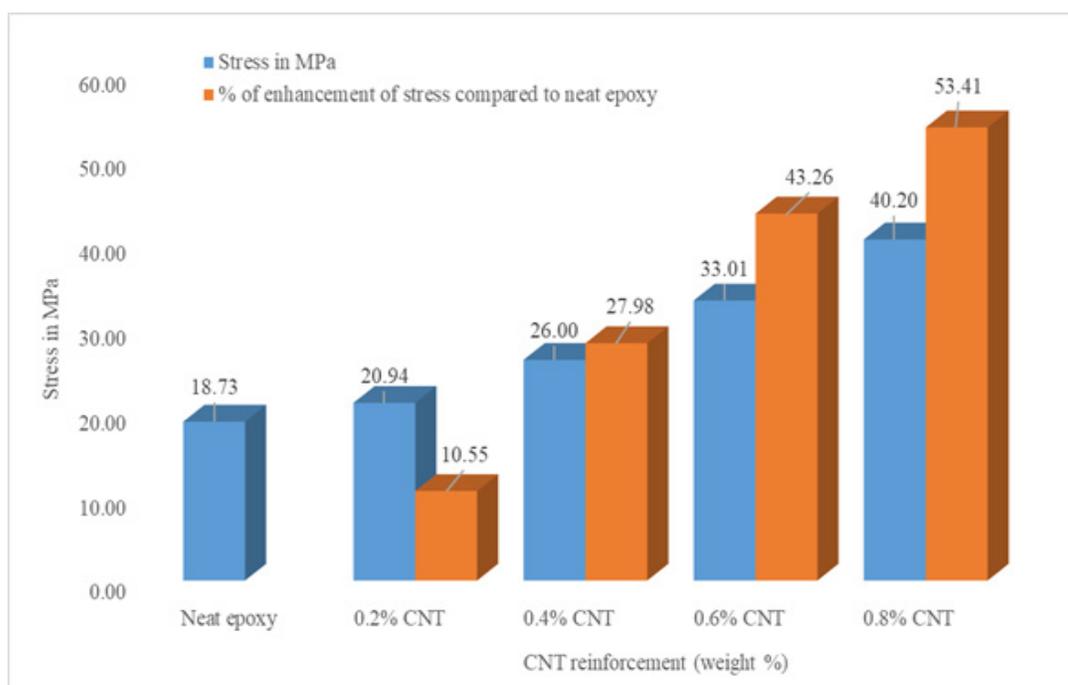


Figure 1. Tensile Strength for various percentage of CNT.

percentage of CNT in epoxy tensile strength is enhanced by 53.41 percentage as compared to neat epoxy. This enhancement is due to epoxy infiltration procedure, the pores of the CNT fibers were shown to be filled with epoxy, and the emergence of covalent bonds, either through the linkage of polymer chains wrapping around the tubes, or even through the emergence of covalent bonds between

the CNT bundles. Interfacial interactions are enhanced. Carboxyl-functionalized nanotubes.

Table 3 is one way ANOVA analysis five independent variable, the p-value which corresponds to the F-Statistic of one-way ANOVA is lower than 0.05. And all are significant as per the Tukey HSD test, Scheffé, Bonferroni and Holm multiple comparison tests.

Table 3. One-way ANOVA of 5 independent treatments

Source	Sum of Squares (SS)	Degrees of Freedom (Nv)	Mean Square (MS)	F Statistic	P-Value
Treatment	1,567.3039	4	391.8260	340.5536	1.1102e ⁻¹⁶
error	23.0111	20	1.1506		
Total	1,590.3150	24			

3.2 Tukey HSD Results

Tukey HSD method used to study the influence and significant levels between each CNT reinforcements.

Table 4 and 5 indicates that by adding 0.2 weight percentage of CNT in epoxy, no such improvements in tensile property. By adding more than 0.2 weight

Table 4. Tukey HSD results.

Treatments pair	Tukey HSD Q statistic	Tukey HSD p-value	Tukey HSD inference	Significant Levels
A vs B	4.6049	0.0288465	p<0.05	Low
A vs C	15.1668	0.0010053	p<0.01	High
B vs C	10.5620	0.0010053	p<0.01	High
B vs D	25.1579	0.0010053	p<0.01	High
A vs D	29.7627	0.0010053	p<0.01	High
C vs D	14.5959	0.0010053	p<0.01	High
C vs E	29.5929	0.0010053	p<0.01	High
D vs E	14.9970	0.0010053	p<0.01	High
A vs E	44.7598	0.0010053	p<0.01	High
B vs E	40.1549	0.0010053	p<0.01	High

Table 5. Scheffe statics analysis results.

Treatments pair	Scheffé TT-statistic	Scheffé p-value	Scheffé inference	Significant Levels
A vs B	3.2561	0.0634629	Insignificant	Very Poor
A vs C	10.7246	4.8419e ⁻⁰⁸	Significant	High
B vs C	7.4684	1.3713e ⁻⁰⁵	Significant	High
A vs D	21.0454	2.3948e ⁻¹³	Significant	Very High
B vs D	17.7893	5.7312e ⁻¹²	Significant	Very High
C vs D	10.3209	9.1774e ⁻⁰⁸	Significant	High
C vs E	20.9254	2.6712e ⁻¹³	Significant	Very High
D vs E	10.6045	5.8462e ⁻⁰⁸	Significant	High
A vs E	31.6499	1.1102e ⁻¹⁶	Significant	Very High
B vs E	28.3938	7.7716e ⁻¹⁶	Significant	Very High

percentage of CNT any appreciable change in tensile properties and its significant levels are same as per Tukey HSD. But according to Scheffe statics test, behind 0.2 weigh percentage of CNT in epoxy significant levels are changes at various percentage.

3.3 Bonferroni and Holm Multiple Comparison

According to Bonferroni and Holm results it is clearly analysed that, by adding CNT from 0.2 to 0.8 weight percentage there is a significant change in tensile property. Further, p-value of Bonferroni and Holm indicates the

magnitude of significance. Table 6 shows the p value, between A to B no much change in the values. It is clearly indicates that, it is having very poor level of significant. By adding 0.4 weight percentage of CNT in epoxy, p value included by $3.3159e^{-06}$, this indicates that medium/moderate influence in tensile strength. Similarly, at 0.6 of CNT, p value improved to $1.8554e^{-08}$, this clearly indicates that enhancement of tensile property. Similarly, Table 7 shows the TT statistic and p value for various percentage of reinforcements. The TT statistic value is 21.0454 for 0.6 percentage of reinforcement, however it is only 31.6499 at 0.8 percentage.

Table 6. Bonferroni and Holm results: all pairs simultaneously compared.

Treatments pair	Bonferroni and Holm TT-statistic	Bonferroni p-value	Bonferroni inference	Levels	Holm p-value	Holm inference	Levels
A vs B	3.2561	0.0395469	Significant	Very Poor	0.0039547	Significant	Very Poor
A vs C	10.7246	$9.6356e^{-09}$	Significant	Medium	$4.8178e^{-09}$	Significant	Medium
A vs D	21.0454	$3.9968e^{-14}$	Significant	Very High	$3.1974e^{-14}$	Significant	Very High
A vs E	31.6499	$0.0000e^{+00}$	Significant	Extremely High	$0.0000e^{+00}$	Significant	Extremely High
B vs C	7.4684	$3.3159e^{-06}$	Significant	Medium	$6.6319e^{-07}$	Significant	Medium
B vs D	17.7893	$9.9698e^{-13}$	Significant	Very High	$5.9819e^{-13}$	Significant	Very High
B vs E	28.3938	$0.0000e^{+00}$	Significant	Extremely High	$0.0000e^{+00}$	Significant	Extremely High
C vs D	10.3209	$1.8554e^{-08}$	Significant	Medium	$5.5661e^{-09}$	Significant	Medium
C vs E	20.9254	$4.6629e^{-14}$	Significant	Very High	$3.2641e^{-14}$	Significant	Very High
D vs E	10.6045	$1.1687e^{-08}$	Significant	Medium	$4.6747e^{-09}$	Significant	Medium

Table 7. Bonferroni and Holm results: only pairs relative to A simultaneously compared.

Treatments pair	Bonferroni and Holm TT-statistic	Bonferroni p-value	Bonferroni inference	Holm p-value	Holm inference	Level
A vs B	3.2561	0.0158188	Significant	0.0039547	Significant	Moderate
A vs C	10.7246	$3.8542e^{-09}$	Significant	$1.9271e^{-09}$	Significant	Medium
A vs D	21.0454	$1.5987e^{-14}$	Significant	$1.1990e^{-14}$	Significant	Very High
A vs E	31.6499	$0.0000e^{+00}$	Significant	$0.0000e^{+00}$	Significant	Extremely High

4.0 Conclusions

The CTN particles reinforced epoxy composites fabricated for various weight fractions, the influence of nanoparticles was statistically determined using ANOVA. Further, Tukey HSD test, Scheffé, Bonferroni and Holm multiple comparison tests are used to study the significance levels for various independent variables. The following conclusions observed,

- The acetone solution helps to get mixing of CNT parties properly in epoxy.
- The mechanical properties of composites enhanced by adding CNT in epoxy.
- The ANOVA method clearly indicated, significant influence between each groups/treatment.
- The Bonferroni, Holm multiple tests and Tukey HSD test are clearly indicating that by adding 0.2 weight fraction of CNT in epoxy, there is no such improvement in mechanical properties. However, by adding 0.4, 0.6 and 0.8 weight fraction of CNT influence significantly.

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