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Studies on Morphological and Mechanical Properties of Epoxy/Vinylester-MWCNT Nanocomposites

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Abstract: This study investigates the mechanical characterization of epoxy/vinylester multi-walled carbon nanotube (MWCNT) nanocomposites, focusing on key properties such as modulus, ultimate tensile strength (UTS), Izod impact strength, dynamic mechanical analysis (DMA), and surface morphology. The incorporation of MWCNTs into the epoxy/vinylester matrix aims to enhance the mechanical performance by leveraging their exceptional strength and stiffness. Tensile tests were conducted to determine the modulus and UTS, providing insights into the reinforcement efficiency of MWCNTs. Izod impact tests evaluated the nanocomposite's resistance to sudden impact loading, while DMA was employed to analyze the viscoelastic behavior, including storage modulus, loss modulus, and damping characteristics across varying temperatures. Surface morphology was examined using scanning electron microscopy (SEM) to assess the dispersion of MWCNTs and their interfacial bonding with the polymer matrix. The results demonstrated significant improvements in mechanical properties, indicating the potential of epoxy/vinylester-MWCNT nanocomposites for high-performance applications in structural and functional materials.

Keywords: Epoxy/vinylester nanocomposites, multi-walled carbon nanotubes, Mechanical characterization, Tensile strength, Impact strength, Dynamic mechanical analysis, Surface morphology.

Introduction

Nanocomposites have gained widespread interest in materials science because they demonstrate enhanced mechanical and functional attributes compared to traditional composites. Among the various reinforcing materials, multiwalled carbon nanotubes (MWCNTs) have proven to be particularly effective due to their outstanding mechanical, thermal, and electrical properties, which arise from their high aspect ratio, excellent tensile strength, and exceptional thermal conductivity [1,2]. Integrating MWCNTs into polymer matrices like epoxy and vinylester resins has been shown to significantly elevate the overall performance of composites, rendering them ideal for high-demand applications in industries such as aerospace, automotive, and structural engineering [3].

Epoxy and vinylester resins, as widely utilized thermosetting polymers, offer strong adhesion, robust chemical resistance, and notable mechanical strength. However, their natural brittleness and limited resistance to impact often require modifications to meet the stringent requirements of advanced applications [4]. Incorporating MWCNTs into these resins has been demonstrated to enhance key mechanical properties, including stiffness, ultimate tensile strength (UTS), density, and hardness, all while maintaining a lightweight profile [5].

This research investigates the mechanical properties of epoxy/vinylester-MWCNT nanocomposites, analysing modulus, ultimate tensile strength (UTS), Izod impact strength, specific gravity, and hardness. Evaluating these properties is essential for optimizing composite formulations and aligning them with specific application needs. The study aims to explore the combined effects of MWCNT inclusion, supporting the advancement of high-performance composite materials.

Epoxy resins, known for their high modulus, strength, electrical insulating properties, and chemical stability, are widely used in structural engineering as adhesives or matrix materials in the aerospace, marine, and automotive industries [6-8]. However, their inherent brittleness, resulting from a highly cross-linked microstructure, limits their broader application in engineering [9-10]. To address this limitation, improving the mechanical properties of epoxy is essential for creating functional composite materials. An effective approach involves incorporating carbon-based nanofillers such as carbon nanotubes (CNTs) [11], silicon carbide (SiC) [12], carbon black (CB) [13], expanded graphite (EG), and graphene nanoplatelets (GNPs) [14] into the epoxy matrix. In addition to enhancing mechanical properties, the inclusion of nanoparticles also improves the thermal [15] and electrical properties [16] of the epoxy phase. Among these, CNTs are particularly favoured for their exceptional mechanical [17], electrical [18], and thermal properties [19].

Carbon nanotubes (CNTs) are nanoscale cylindrical structures made entirely of carbon atoms. They are well-known for their outstanding mechanical [17], electrical [18], and thermal properties [19]. CNTs can be classified into three main types: single-walled carbon nanotubes (SWCNTs), consisting of a single graphene sheet rolled into a cylinder; double-walled carbon nanotubes (DWCNTs), formed by two concentric SWCNTs; and multi-walled carbon nanotubes (MWCNTs), made up of multiple graphene cylinders nested within each other. These materials are widely used in CNT/polymer composites for structural and functional applications [20-21].

Carbon black (CB), a product of the incomplete combustion of carbonaceous or petroleum-based materials, is widely recognized as a primary reinforcing filler in the polymer industry [22, 23]. The particle size of CB typically ranges from 5 to 100 nm, but it often forms aggregates that can reach sizes up to 500 nm. The mechanical reinforcement effects of carbon-based nanoparticles depend on factors such as morphological structure, particle size, distribution uniformity, and the volume fraction of nanoparticles within the epoxy matrix [19]. CBs, carbon nanotubes (CNTs), and expanded graphite (EG) can be classified as 0D, 1D, and 2D materials, respectively, based on their morphological structures. The surface area of CNTs, for instance, provides an effective interface for stress transfer from the epoxy matrix to the nanoparticles. Greater interfacial areas between the matrix and the nanoparticles enhance load transfer capability, improving the overall mechanical performance of the composite [24].

Experimental

A liquid epoxy resin (Diglycidyl ether of bisphenol A) that comprises for more than 75% of the resin exploited in industrial applications, is the epoxy resin employed in the current research. It is provided below the skill name Araldite® GY 2600, which is a registered brand for the commercial resins manufactured by Huntsman Advanced Materials. Polyamines or their adducts can cure Araldite® GY 2600 to produce solvent-free coatings, flooring screeds, trowelling compounds, etc. Epoxy resins and unsaturated polyesters share chemical properties with vinyl esters. They were developed as a combination of the two materials, offering both the ease of application and cost-effectiveness of polyesters, along with the mechanical and thermal properties of epoxies.

MWCNTs from the NANOCYLTM NC7000 series, which were synthesized using the catalytic carbon vapor deposition (CCVD) method. These are particularly important in applications that require a low electrical separation threshold, such as high-performance electrostatic dissipative coatings. The NC7000 powder is available in quantities ranging from 2 kg to several tonnes, with pre-dispersed forms (PLASTICYLTM, EPOCYLTM, AQUACYLTM) also available

Synthesis of Epoxy-MWCNTs Nanocomposites: MWCNTs and Epoxy resin were mixed in various weight ratios. In order to spread the MWCNTs, the MWCNTs were first added directly to the molten Epoxy resin and then the blend was combined under a magnetic agitator for 10 minutes at 80°C. When the MWCNTs were properly dispersed, we added the hardener, 14.4 gram of aradur 2958, while stirring, and produced several samples. For the first sample preparation in this example, 80 gm of epoxy resin and 14.4 gm of hardener was applied. Second, the same volume of epoxy resin and hardener has been used; the only variation was indeed the percentage of MWCNTs, which varied between 0.5, 1.0, and 2.0. After that, a casting method was used to prepare the samples with the correct dimensions before the molten sample was allowed to form into the aluminium mould. The sample was then removed out of the mould and left at ambient temperature for 72 hours for post-curing.

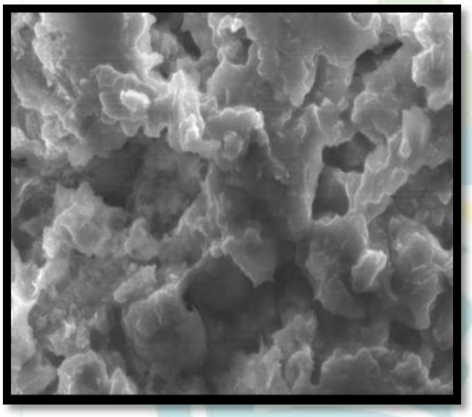
Synthesis of Vinyl ester-MWCNTs Nanocomposites: The Fiberbond 701 resin (Ruia Chemical Pvt. Ltd.) Vinyl Ester matrix used in this research was cured with the hardener (catalyst) "MEKP" and Accelerator (cobalt naphthenate). The MWCNTS was used to produce the nanocomposites. Thus far, the MWCNTs have improved the viscosity of the vinyl ester resin. The dispersion technique was modified in order to increase the maximum CNT content and advance the quality of the nanocomposites. Furthermore, the material could no longer be degassed before curing, which resulted in trapped air in the finished composites. Given that the shear forces generated from mixing in the suspension did, in fact, directly affect the viscosity. As mentioned, I used 30 g of vinylester resin and 0.45 gram each of hardener and accelerator respectively.

Table 1: Specimen Composition.

Sample code	Specimen Composition
1	Epoxy+Hardener+ 0%MWCNT
2	Epoxy+Hardener+0.5%MWCNT
3	Epoxy+Hardener+1% MWCNT
4	Epoxy+Hardener+2% MWCNT
5	Vinylester+Hardener+Accelerator
6	Vinylester+Hardener+Accelerator+0.5% MWCNT
7	Vinylester+Hardener+Accelerator+1%MWCNT
8	Vinylester+Hardener+Accelerator+2%MWCNT

Results and Discussions

Morphological analysis: Scanning Electron Microscopy (SEM) images of of Epoxy Vinylester-MWCNT nanocomposites are shown in Figure 1. The MWCNT was shown as a nano-ribbon with a width of around 200 nm and a length from several microns in the SEM image illustrated in Fig. 1. It was obvious to anticipate just how form of nanoribbon would interlock and entangle with the matrix's polymer chains if it were been the across. For ease of comparison, all Samples from the one of MWCNT—were focused at a magnification of 10,000 times, except for the image of MWCNT. The cleavage surface of the 2 wt% MWCNT-epoxy sample displayed significant differences compared to the pure epoxy sample, as shown in the SEM images in Figures 4.26 B, C, and D. The fracture surface revealed particles with distinct, white fracture lines. In general, as seen in figures 1 C and D, the SEM images of the 2 wt% MWCNT epoxy samples demonstrated a tendency for the cleavage surface to fracture with smaller, more irregular fracture zones. In a specific instance, it emerged that tiny white lines that were considered to represent MWCNT (the nanoribbon) appeared from the fracture surfaces. As a result, the MWCNT that was added to the epoxy performed like the grid lines of a net. According to a convergence of SEM images with the findings of a tensile test, the insertion of this type of "nanoribbon" into the epoxy matrix would enable the nanoribbon to interlock with the epoxy network chains. More MWCNT content seems to be helping the interlocking mechanism more effectively prevail. As a result, it appeared that introducing more of this variety of MWCNT would enable the sample to be become brittle, or, to put it another way, that the ductility would decrease as more of this form of MWCNT was introduced. The samples' brittleness rises with the quantity of MWCNT applied to the epoxy. As a result, the epoxy chains system was impeded by the ribbon-like MWCNT from combining properly, producing the MWCNT epoxy samples more brittle, but it also rendered the samples stronger and stiffer to a certain extent.



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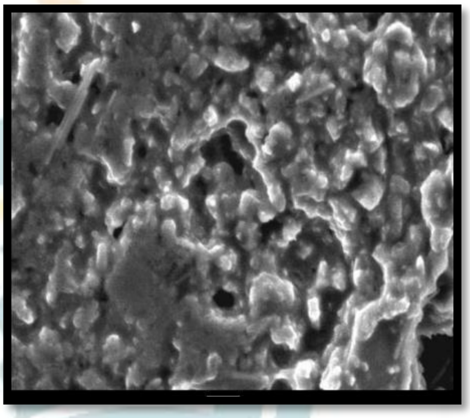

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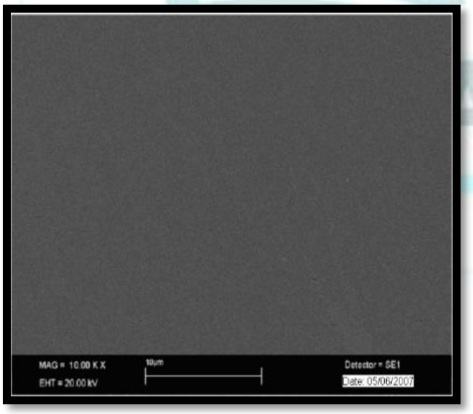

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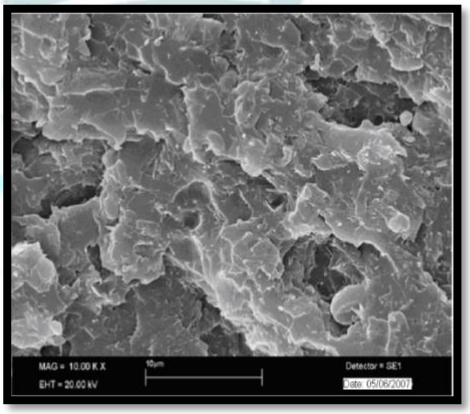

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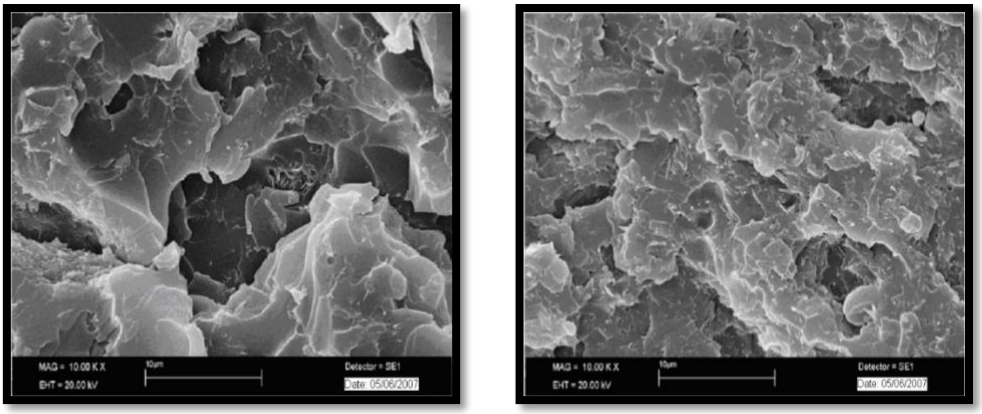


(e) **(f)**

Figure 1: Scanning Electron Microscopy (SEM) of (a) Pure Epoxy, (b) Pure vinylester+Hardener, (c) Epoxy+Hardener, (d) Epoxy+Hardener+2% MWCNT, (e) VE+Hardener+Accelerator+0.5% MWCNT and (f) VE+Hardener+Acclerator.

Impact Properties: The "Digital Pendulum Impact Tester (Ceast Instruments Ltd. P/N 6963 Italy)" explores ways nanoparticles influence impact strength. The preparation and analysis of nanocomposite specimens was performed in accordance with "ASTMD-256." Impact testing was performed using a pendulum-type device following the Izod method, ASTM D-256 (ERF 33-82), where the test specimen was established as a cantilever beam with the notched side facing the striker. Five illustrations of each preparation (specimen dimension in length: 63.5 mm, width: 12.7 mm, depth: 3 mm) are examined, and the result obtained is listed as the notched Izod impact strength (Fig. 2). Evaluating impact strength is a crucial element in material evaluation programs. Although many of the testing methods are relatively simple, interpreting the results can be complex because impact strength is not a single, intrinsic property but a composite of several factors. The Izod impact strength test, defined by ASTM-D256, is a standard procedure used to measure this property. In (Fig. 2a) Vinyl Ester-MWCNT Nanocomposite durability and resilience to environmental breakdown of vinyl ester resins are widely recognized. Multi-walled carbon nanotubes (MWCNTs) improve energy dissipation and fracture deflection, which increases impact resistance. Impact strength can increase as a result of improved interfacial bonding or fall as a result of agglomeration, depending on MWCNT dispersion and loading. In (Fig. 2b) Epoxy-MWCNT Nanocomposites as compared to vinyl esters, epoxy resins are typically more brittle despite having a higher degree of rigidity. MWCNTs can increase energy absorption and connect microcracks to increase impact strength. MWCNT dispersion, surface functionalization, and contact with the epoxy matrix all affect the final result.

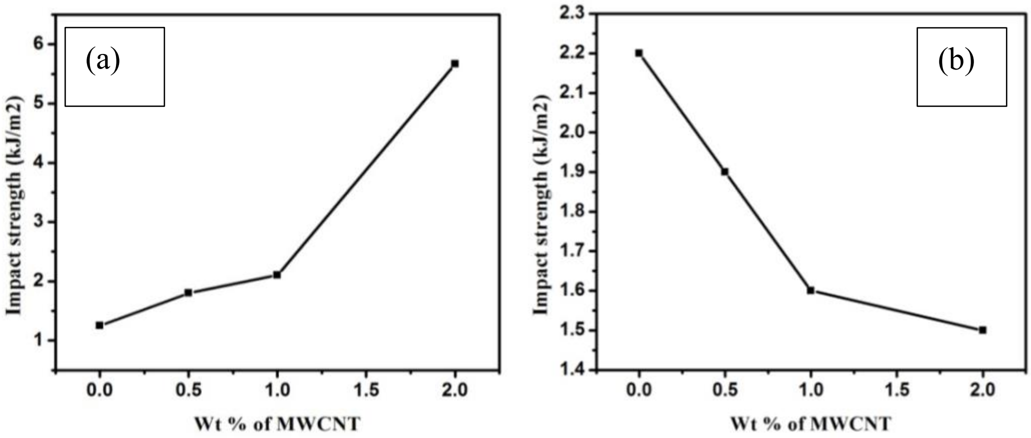
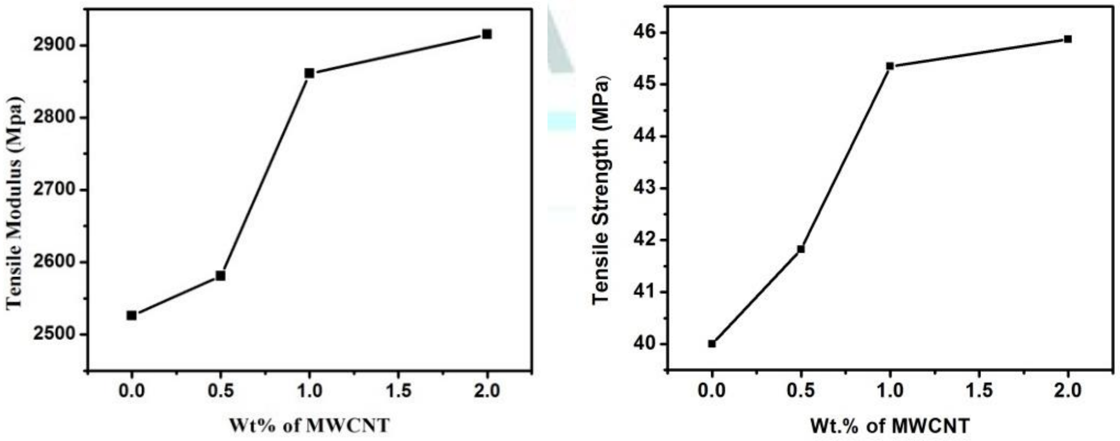


Figure 2: (a)Vinyl ester-MWCNT Nanocomposites impact strength (b) Epoxy-MWCNT Nanocomposites impact strength

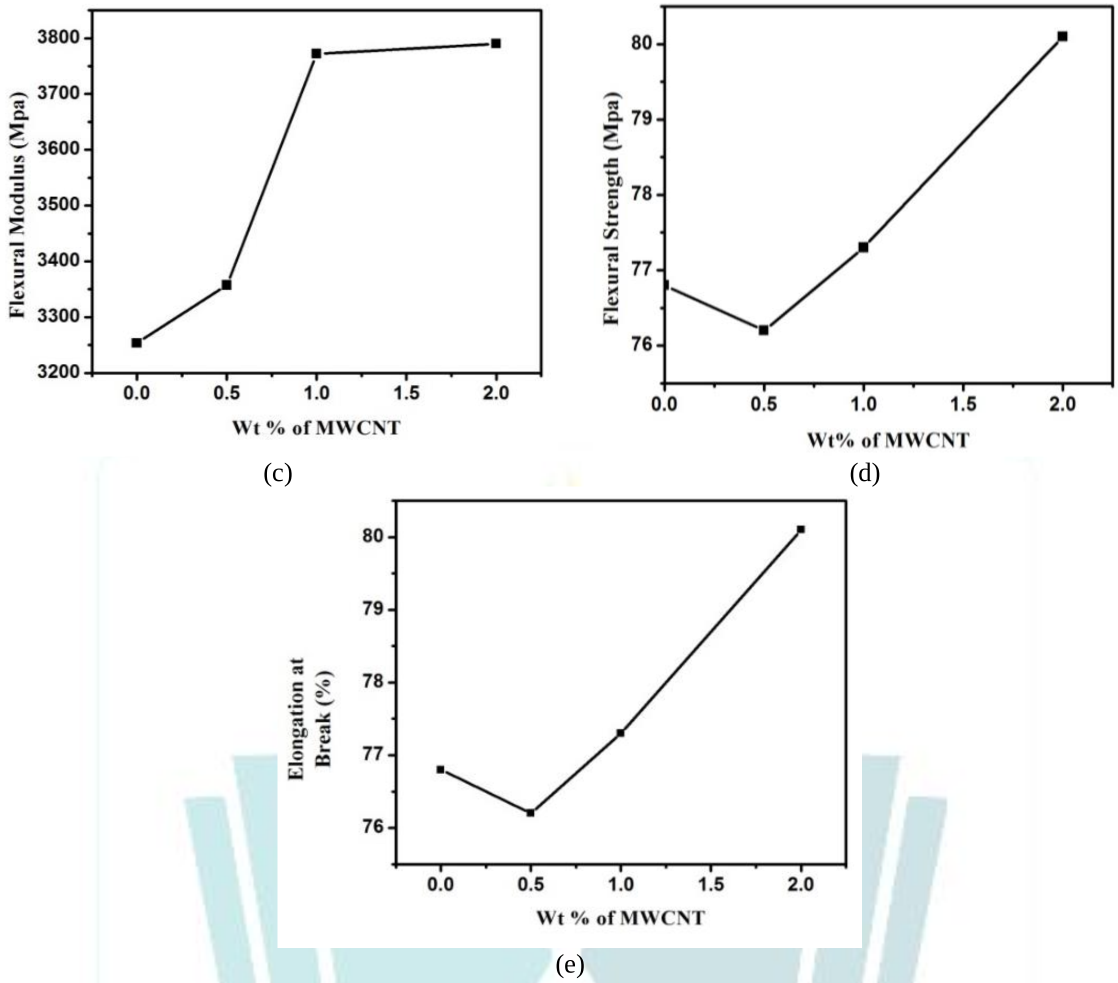
Mechanical properties: When MWCNT is introduced to an epoxy resin system, the mechanical behaviour, in particular the elastic modulus, improves. It is observed that the elastic modulus growths with cumulative MWCNT content. The modulus improves by 25% only with 1 wt.% of MWCNT and so by 50% for 2 wt.%. The MWCNT particles' excellent dispersion and exfoliation are to responsible for the improvement in elastic modulus. An increased modulus is a result of better dispersion and increased adhesion between the MWCNT particles and the epoxy matrix, which lowers the mobility of the epoxy chains under stress. The formation of a nanocomposite improves the epoxy resin's modulus and flexural strength. Tensile strength is expected to increase up to 1 wt.% MWCNT before reducing after that. It has been observed that the extent of exfoliation affects the increase in modulus. With the same wt.% of MWCNT added, we observed that an exfoliated nanocomposite advances its elastic modulus more than a intercalated nanocomposite. Furthermore, compared to glassy nanocomposites, flexible nanocomposites exhibit a significant increase in flexural strength and modulus. An epoxy vinyl ester-MWCNT nanocomposite's elastic modulus will increase more if it is measured above the glass transition temperature. It has been shown that elastomeric polymers with sub ambient glass transition temperatures can develop mechanical properties significantly, while glassy polymers with high glass transition temperatures only ever see small benefits. The mechanical properties of diverse weight% (0.5,1,2) of the Epoxy/Vinylester-MWCNT nanocomposites are represented on Table 2.

Table 2: Mechanical Data of Epoxy/vinylester-MWCNT Nanocomposites.

Specimen Composition	Tensile Strength (MPa)	Tensile Modulus (MPa)	Flexural Strength (MPa)	Flexural Modulus (MPa)	Elongation at Break (%)	Impact strength (kJ/m^2)
1	40	2524	76.6	3251.25	3.32	2.41
2	42	2578	76.08	3356.32	3.34	2.20
3	45.5	2864	77.49	3771.30	3.18	1.67
4	45.7	2911	80.09	3788.22	3.15	1.58
5	7.76	2278.02	32.89	3001.59	0.48	1.28
6	17.11	2592.16	44.07	3752.44	0.87	1.84
7	11.43	2521.35	47.18	3801.81	0.52	2.18
8	6.8	2325.38	55.41	4914.69	0.46	5.69



(a) (b)

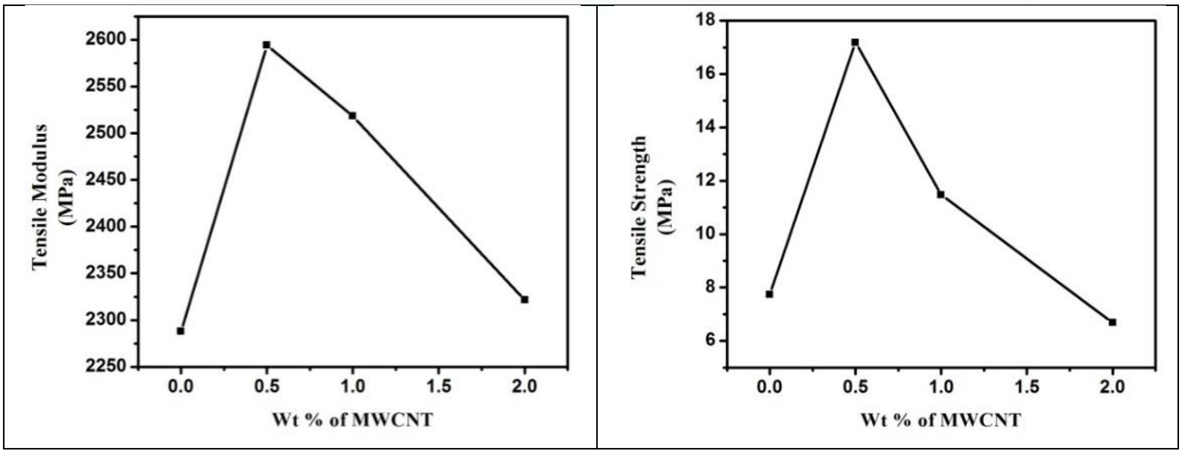


(c) (d)

(e)

Figure 3: The resulting mechanical properties in different composition of MWCNT/ Epoxy **(a)** Tensile modulus, **(b)**Tensile Strength, **(c)** Flexural modulus, **(d)** Flexural strength, **(e)** Elongation at Break.

Tensile testing was used to assess the synthesized nanocomposites' mechanical characteristics in (Fig. 3 (a-e) show the tensile behaviour of the samples under examination. The Young's modulus increased with decreasing multiwall carbon nanotube (MWCNT) concentrations. However, the presence of MWCNTs had a more pronounced negative impact on the modulus at higher concentrations. Strong interactions with the low-viscosity, accelerated, aliphatic polyamine hardener contribute to their improved dispersibility in the polar epoxy resin, which is responsible for this phenomenon. When 2 weight percent MWCNT was added, the Young's modulus rose from 3.29 GPa (± 0.10 GPa) for the pure resin to 3.50 GPa (± 0.11 GPa).



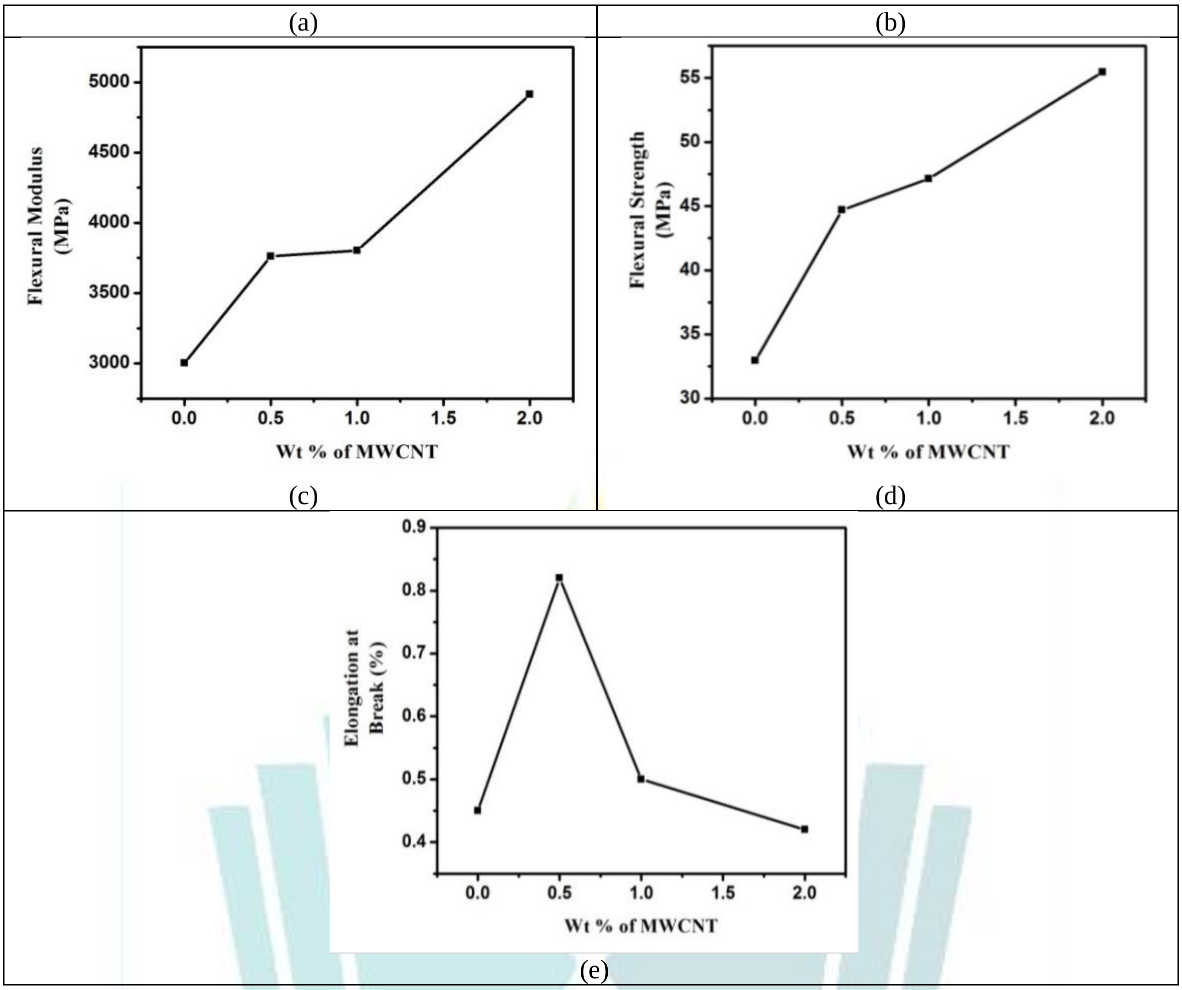


Figure 4: Mechanical properties of MWCNT/Vinylester: **(a)** Tensile modulus, **(b)**Tensile Strength, **(c)** Flexural modulus, **(d)** Flexural strength, **(e)** Elongation at Break.

It Vinyl Ester-MWCNT nanocomposites calculates the highest stress a material can bear before disintegrating when deformed. In illustrates (Fig.4 c & d) the material's resistance to deformation and its stiffness in bending. MWCNTs increase flexural strength using improving load transmission. Better resistance to bending stress is an effect of improved interfacial bonding between vinyl ester and MWCNTs. The modulus rises with uniform MWCNT dispersion because of the increased stiffness; however, insufficient dispersion could end up in lower performance and stress concentration. In Epoxy-MWCNT Nanocomposites reinforcement, which distributes stress and bridges microcracks, provides a strength higher than apparent epoxy (fig.3 c & d). Since of the stiff nature of nanotubes, it usually rises as the MWCNT content does. While agglomeration can result in defects, proper dispersion improves mechanical characteristics. Flexural attributes are further increased by functionalized MWCNTs, which enhance matrix interaction.

Conclusion

A system of Epoxy/Vinylester-MWCNT nanocomposites underwent comprehensive thermo-mechanical testing. The main focus of the work was on the effects of MWCNT type and integration on the mechanical and morphological characteristics of the nanocomposites. It examined the creation and properties of nanocomposites reinforced with multi-walled carbon nanotubes (MWCNTs) derived from epoxy and vinylester matrices. To evaluate their performance, these composites underwent a thorough thermo-mechanical investigation. The Epoxy/Vinylester-MWCNT composite performed better in every way because of its increased flexural strength, reduced density, and larger d-spacing (24.2). While silane treatment of the carbon nanotubes (CNTs) further improved the flexural characteristics of carbon/CNT/epoxy composites, the addition of CNTs increased the strength and flexural moduli of carbon/epoxy composites. In particular, as compared to their acid-treated counterparts, the silane-treated carbon/CNT/epoxy composite showed flexural moduli and strengths that were 34% and 20% greater, respectively. The carbon/epoxy composites' resistance to wear was further enhanced by the inclusion and surface treatment of carbon nanotubes. The higher flexural and wear resistance of the carbon/CNT/epoxy multiscale composites was a result of the silane-modified MWCNTs' higher dispersibility and improved interfacial contact with the epoxy matrix. The tensile strength, flexural strength, tensile modulus, and flexural modulus of 0.5% Vinylester-MWCNT reinforced epoxy nanocomposites and 2% MWCNT with Epoxy are improved by 4%, 6%, 19%, and 23%, respectively. The mechanical and physical characteristics of the cured epoxy matrix are determined by the chemical reaction between the particles and epoxy

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