



Article Enhanced Wear Resistance in Carbon Nanotube-Filled Bio-Epoxy Composites: A Comprehensive Analysis via Scanning Electron Microscopy and Atomic Force Microscopy

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Abstract: This investigation focuses on the wear resistance and surface morphology of multi-walled carbon nanotube (MWCNT)-filled bio-based epoxy composites. This study examines the impact of different MWCNT concentrations (0 Wt.%, 0.25 Wt.%, 0.50 Wt.%, and 0.75 Wt.%) on the wear properties of these composites. Techniques such as scanning electron microscopy (SEM) and atomic force microscopy (AFM) were utilized for comprehensive surface characterization. The results demonstrated a direct correlation between the MWCNT content and the wear resistance of the composites, which were corroborated by robust statistical analysis. Furthermore, SEM and AFM observations disclosed incremental enhancements in both wear resistance and surface quality as the MWCNT concentration increased. This research not only augments the understanding of wear mechanisms in bio-based epoxy composites but also aligns with the burgeoning focus on sustainable materials.

Keywords: CNT-filled bio-epoxy composites; wear resistance; surface morphology; scanning electron microscopy (SEM); atomic force microscopy (AFM)

1. Introduction

Carbon fiber-reinforced composites, formed by amalgamating carbon fibers with polymeric matrices, have gained considerable attention across multiple industries. Their superior physical and mechanical attributes make them particularly useful in the automotive and aerospace sectors [1,2]. Factors such as the type, shape, direction, and content of the reinforcement, as well as the composition of the matrix, influence the properties of these composites [3]. Carbon fibers, when integrated as reinforcements in thermoplastic polymers like short fibers and woven fabrics, have been demonstrated to enhance wear resistance. For example, incorporating approximately 20% short carbon fibers into polyetherimide significantly reduces friction and improves wear resistance [4,5].

Moreover, fabric-based fiber reinforcement utilizes the graphite structure in carbon fibers to develop self-lubricating bearings. Numerous studies have investigated the effects of fiber volume and orientation on the abrasion and frictional properties of carbon fiber composites [6]. Epoxy resins, renowned for their processing characteristics, high strength, and resistance to temperature and solvents, are increasingly used in technical applications.



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However, their three-dimensional cross-linked bond structure limits their applicability in tribological settings compared to thermoplastics [7]. To address this limitation, fillers are added to produce wear-resistant composite materials, enabling the use of epoxy in high-wear environments [8]. Amid growing environmental concerns, there is a shift towards bio-based resins as alternatives to traditional fossil fuel-based epoxies [9]. Although they currently occupy a small market share, bio-based polymers offer a sustainable path forward [10]. One of the key challenges researchers face is enhancing the properties of partially or fully bio-based epoxy polymers by incorporating appropriate reinforcements. There is a noticeable gap in comprehensive research exploring the mechanical and tribological properties of bio-based epoxy composites reinforced with carbon nanotubes (CNTs) [11]. The existing literature on CNT-reinforced composites provides fragmented data on their mechanical properties. This inconsistency is often attributed to the complex interfacial bonding between the matrix and CNTs during the composite mixing process [12–15]. Recent advancements in materials science have led to the development of nanocomposite materials with unique and improved properties. The inclusion of CNTs into polymer matrices has become a focal point due to their exceptional mechanical, electrical, and thermal characteristics. Understanding the wear behavior of such nanocomposites, particularly in dry sliding wear conditions, is crucial for enhancing their performance and broadening their applications [16,17]. In tribological applications, the coefficient of friction is just as crucial a property as wear characteristics. In macroscale friction and wear tests, it has been widely accepted that CNTs undergo a structural change on sliding surfaces, replacing irregularly shaped carbon and typically yielding a coefficient of friction value of approximately 0.2 [18]. This research article aims to investigate the dry sliding wear behavior of bio-epoxy nanocomposites filled with CNTs. Bio-epoxy, a bio-based epoxy resin, serves as an eco-friendly and sustainable alternative to conventional petroleum-based epoxy resins. It was chosen as the matrix material in this study due to its favorable mechanical properties and compatibility with CNTs.

2. Background Theory

Bansal et al. [19] focused on enhancing the mechanical properties of epoxy-based composites using a small percentage (0.25 Wt.%) of carbon nanotubes (CNTs) as reinforcement. The research involved a solution-mixing method, dispersion of CNTs in a solvent using a probe sonicator, and curing with an amine-based agent. The resulting nanocomposite was analyzed for morphology and nanoindentation for mechanical properties using scanning electron microscopy (SEM). The study found that even with this low CNT reinforcement, the epoxy–CNT composite showed a substantial 36% increase in modulus and a 16% increase in hardness compared to pristine epoxy, highlighting the potential for improving mechanical properties with CNTs. Kasemsiri et al. [20] introduced innovative shape-memory polymers (SMPs) capable of reshaping through bond exchange reactions (BERs). The researchers developed polymer composites using epoxy phenolic novolac (EPN) and bio-based cashew nutshell liquid (CNSL) reinforced with multi-walled carbon nanotubes (CNTs). These composites displayed shape memory and self-welding properties, allowing them to change shape via BERs. The research investigated their shape-memory mechanisms, including photo-induced shape memory induced by near-infrared (NIR) light. The composite with 0.3 Wt.% CNTs exhibited the best shape-memory performance. The study highlighted the potential for advanced applications in smart materials due to shape memory, reconfiguration, and self-welding properties in these EPN/CNSL composites. Chen et al. [21] worked on creating a novel material, HBPPA-CNT, by combining carbon nanotubes (CNTs) with reactive hyperbranched polyphosphoramide (HBPPA). This hybrid enhances the flame-retardant and mechanical properties of epoxy composites. The composite shows impressive flame-retardant performance, mechanical strength improvement, and reduced heat release rates. This approach holds promise for expanding the use of carbon materials in polymer composites.

Tripathi et al. [22] developed lightweight bio-composites with damage-sensing capabilities by incorporating conductive polymer nanocomposite-based quantum resistive sensors (sQRSs) into flax fiber-epoxy composites. The sQRSs monitored structural health and predicted potential failures. The study found a strong correlation between sQRS signals and mechanical behavior, aiding in assessing structural changes and damage likelihood in the bio-composite. This research enhances the understanding of flax fiber-based composites' mechanical behavior, addressing challenging fibril assembly issues. Han et al. [23] addressed the challenge of achieving high-performance nanocomposites with well-dispersed and aligned nanometer-sized components, such as carbon nanotubes (CNTs). The researchers developed a bio-inspired strategy to control CNT aggregation, limiting it to sub-20–50 nm dimensions. This controlled aggregation, combined with a multistep stretching process, led to CNT-polymer (bismaleimide) composites with exceptional tensile strength (up to 6.94 GPa) and toughness (up to 192 MPa). These properties exceeded those of carbon fiber/epoxy composites by more than 100%. The study's approach has the potential to be applied in developing multifunctional and smart nanocomposites, enabling efficient transfer of mechanical, thermal, and electrical signals across nanoscale components. Lachman and Wagner [24] researched the analysis of carbon nanotube (CNT) composites. XPS analysis confirms the presence of carboxylic and amino groups on CNTs. Well-dispersed pristine CNTs and carboxylated CNTs significantly enhance Young's modulus, while aminated CNTs have a minor effect. Aminated CNTs do not weaken tensile strength, unlike other CNT composites. Toughness measurements show mixed results, with no significant improvement in impact toughness due to high strain rates. However, fracture toughness (K_{IC}) is notably higher in CNT-based composites, particularly well-dispersed aminated CNTs, indicating excellent interfacial bonding. SEM analysis shows strong CNT-epoxy adhesion, suggesting reinforcement beyond the CNTs' initial volume fraction. Pull-out length measurements confirm the critical length concept in nanocomposites and reveal that increased interfacial adhesion leads to higher toughness. Varghai et al. [25] investigated bio-based epoxy nanocomposites containing untreated multi-wall carbon nanotubes (MWCNTs) and a bio-based epoxy called DGEDP-Bu. The authors compared the properties to commercial bisphenol A (DGEBA) epoxy composites. Both epoxy types exhibited percolation thresholds for electrical and rheological properties at 0.05 Wt.% and 0.2 Wt.% MWCNT loading. DGEDP-Bu composites showed equal or better properties than DGEBA composites. With 0.2 Wt.% MWCNTs, DGEDP-Bu composites had significantly higher electrical conductivity and rheological yield stress. Rheological analysis confirmed the formation of continuous MWCNT networks in both epoxy types between 0.1 and 0.2 Wt.% MWCNT loading. DGEDP-Bu demonstrated equal or superior mechanical performance and improved electrical conductivity compared to DGEBA, particularly with MWCNT loading. Huang et al. [26] used soy protein isolate (SPI), derived from soybeans, as a biosurfactant to treat carbon nanotubes (CNTs) in nanocomposite production. SPI has proven to be highly effective in improving dispersion, electrical conductivity, and mechanical properties. Compared to sodium dodecyl sulfate (SDS), which is a conventional surfactant, SPI efficiently functionalizes CNTs, reducing agglomeration and stabilizing particle sizes. The SPI-CNT/epoxy nanocomposite exhibits a substantial six-order magnitude increase in electrical conductivity at 0.5 Wt.% CNT loading compared to pure epoxy, surpassing pristine CNTs/epoxy and SDS-treated counterparts. Additionally, mechanical properties, including tensile modulus, strength, and fracture toughness, significantly improve with SPI-CNTs, outperforming pristine CNTs and SDS-treated CNTs. In situ tensile tests show that SPI-CNTs effectively halt crack propagation, making SPI a promising bio-based additive for enhancing polymeric nanocomposites. Zeng et al. [27] investigated the impact of gelatin-modified carbon nanotubes (g-CNTs) on carbon-fiber-reinforced polymer (CFRP) composites. The results showed that g-CNTs enhanced the fiber/matrix interface and overall mechanical performance. A 40.3% increase in interfacial normal strength (IFNS) and a 22.1% to 25.3% improvement in flexural strength/modulus were observed in CFRP composites.

Introducing g-CNTs at 0.1 Wt.% also improves IFNS by 12.6% and flexural strength/modulus by 20.3%/11.4%. This study lays the groundwork for advanced CNTreinforced CFRP composites. The study of bio-epoxy and carbon nanotubes (CNTs) is significant for wear applications and surface finish improvement. This research addresses the need for advanced materials capable of withstanding wear, friction, and abrasion while maintaining a smooth and durable surface [28]. One key importance of this study lies in the substantial improvement of mechanical properties achieved by incorporating CNTs into bio-epoxy composites. These enhancements include increased hardness, strength, and modulus, making these composites promising candidates for wear-resistant components [29]. Moreover, bio-epoxy–CNT composites offer an exceptional surface finish. This smooth and wear-resistant surface is crucial in applications involving contact, reducing friction and prolonging material lifespan. These composites also exhibit impressive wear resistance, making them suitable for sectors like the machinery, aerospace, and automotive industries. They can extend the operational lifespan of components, leading to cost savings [30,31]. Unique properties such as shape memory and self-welding capabilities enable these composites to recover their shape after deformation, reducing surface irregularities caused by wear and tear. In addition to mechanical benefits, bio-epoxy-CNT composites contribute to energy savings, particularly in applications with high friction. They enhance energy efficiency and reduce operational costs. These materials are biocompatible, making them suitable for medical wear applications like orthopedic implants. They maintain a smooth and biocompatible surface, ensuring patient safety [32,33]. Lastly, compliance with environmental and safety regulations is crucial. Bio-epoxy-CNT composites align with these regulations while offering improved performance, extended component lifespan, reduced maintenance costs, and enhanced environmental sustainability and safety.

3. Materials and Method

3.1. Bio-Based Epoxy

This study utilized FormuLITE, a bio-based epoxy, as the primary material. FormuLITE is an amine-cured epoxy solution designed for high-performance resin matrices derived from renewable resources. Both the resin and the amine hardener are synthesized from CNSL (cashew nutshell liquid), which is a renewable resource. This composition offers multiple benefits, such as low viscosity, efficient wetting capabilities for various reinforcements, extended pot life, balanced mechanical properties, and resistance to heat, water absorption, acid, and alkali solutions [34–36]. FormuLITE epoxy systems are designed for medium-to-large composite part manufacturers and are compatible with techniques like wet lay-up, resin transfer molding (RTM), lamination, and vacuum infusion. Their user-friendly nature also makes them suitable for DIY enthusiasts seeking professional results. Table 1 details the general properties of the bio-based epoxy used in this study.

Table 1. General properties of bio-based epoxy considered for this study.

Parameter	FormuLITE	
Calculated bio-content	36.6	
Mix ratio by weight	100:30	
Mix ratio by volume	100:36	
Mix viscosity at 25 °C (cPs)	700	
Mix viscosity at 40 °C (cPs)	242	
Pot life at 25 °C (min)	105	
Pot life at 40 $^{\circ}$ C (min)	57	
Tg (°C)	92	
Tensile strength (MPa)	62	
Tensile modulus (MPa)	2615	
Elongation at Fmax (%)/Elongation at break (%)	4.8/6.4	
Flexural strength (MPa)	92	
Flexural modulus (MPa)	2262	

3.2. Carbon Nanotubes

Carbon nanotubes (CNTs) were another essential material used in this study. CNTs are formed by arranging carbon atoms in a hexagonal lattice, resulting in cylindrical structures with lengths of several centimeters and nanoscale diameters. Their exceptional mechanical, electrical, and thermal properties make CNTs a promising material in the nanotechnology sector. CNTs can be categorized into two main types: single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs). The MWCNTs used in this study consisted of several concentric layers of carbon atoms, forming a multilayered structure. In contrast, SWCNTs are composed of a single layer of carbon atoms rolled into a seamless cylinder. The properties of the MWCNTs are summarized in Table 2. The stages involved in the preparation of the specimen are illustrated in Figure 1.

Parameter Values $\approx 120 \text{ nm}$ Typical diameter Typical length Up to 1 mm Aspect ratio 4000 900 GPa Elastic modulus Tensile strength 45 GPa Thermal conductivity at 300 K $2500 \text{ W}/(\text{m}\cdot\text{K})$ The smallest effective dose for an anti-static additive 0.5% Homogenizer Carbon Nano Tube Dessicator (CNT) To Vaccum Pump חחב 4112 Variation of Speed 500RPM - 1200RPM per minute cycle Resin Part A FormuLite 2501 A Addition of CNT Homogenization Degasification Hardener Part B Mixture of Part A + Part B+ CNT FormuLite 2401 A Part A + CNT Addition of Hardene Magnetic Stirring Pour into Mould Cavity

Table 2. General properties of CNTs used in this study.

Figure 1. Stages in the specimen preparation.

3.3. Wear Test Specimen Preparation

The wear test specimens were prepared following the ASTM G99 standard [37] for pin-on-disc wear tests. A wooden mold cavity with an 8 mm diameter was created for this purpose. The resin solution was formulated with a 100:30 epoxy-to-hardener mixture, as specified in Table 1. Desired weight percentages of CNT (0 Wt.%, 0.25 Wt.%, 0.50 Wt.%, and 0.75 Wt.%) were carefully mixed with the epoxy. The mixed resin and CNT mixtures were then poured into wooden mold cavities. Techniques such as slow pouring, vibrating the mold, and using a vacuum chamber (MDC Precision, Hayward, CA, USA) were employed to avoid air bubble introduction. Fourier-transform infrared (FTIR) spectroscopy (Make: PerkinElmer, Waltham, MA, USA) was conducted to identify any unidentified components in the CNT powder. The prepared samples were then subjected to wear tests following the Taguchi design of experiments. Subsequent analyses of the wear surfaces were performed using scanning electron microscopy (SEM) (ZEISS GeminiSEM 360) and atomic electron microscopy (AFM) (Hitachi AFM5500M), as supported by the statistical methods cited in [38].

4. Results and Discussion

4.1. Characterization of Carbon Nanotubes

Figure 2 displays the SEM image of the multi-walled carbon nanotubes (MWCNTs) used in this study, offering a detailed view of the tube dimensions. This image provides valuable insights into the size and morphology of the CNTs, which are crucial for understanding their interaction with the bio-epoxy matrix. Further characterization was carried out using Fourier-transform infrared (FTIR) spectroscopy. The FTIR spectrum depicted in Figure 3 shows four significant peaks at specific wavenumbers: 3249.1 cm⁻¹, 2801.3 cm⁻¹, 2146 cm⁻¹, and 1342 cm⁻¹. These peaks correspond to various functional groups and chemical bonds, providing insights into the chemical composition of the MWCNTs.



Figure 2. High-resolution SEM image of the carbon nanotubes.



Figure 3. High-resolution FTIR spectrum of CNTs.

The peaks in the FTIR spectrum reveal the presence of specific functional groups. The peak at 3249.1 cm⁻¹ corresponds to the stretching vibration of the O-H bond in carboxyl groups, indicating the presence of oxygen-containing groups on the surface of the MWCNTs. The peak at 2801.3 cm⁻¹ is associated with the O-H stretching in highly hydrogen-bonded -COOH groups, further confirming the functionalization of the MWCNTs. The peaks at 2146 cm⁻¹ and 1342 cm⁻¹ are linked to the stretching vibrations of the -C=O bond in the carboxyl groups and the carboxylate anion (-COO-) group, respectively.

4.2. Functionalization and Surface Properties

The peaks observed in the FTIR spectrum signify the formation of oxygen-containing functional groups on the surface of the modified MWCNTs, which occur during the acidic oxidation process. These functional groups not only enhance the hydrophilicity of the carbon nanotube surface but also provide multiple adsorption sites. This results in improved adsorption capacity, making the MWCNTs more compatible with various matrices, including bio-based epoxy. The specific surface area of the MWCNTs was determined using the Brunauer–Emmett–Teller (BET) method and was found to be $63.17 \text{ m}^2/\text{g}$ [39]. This high surface area indicates that the MWCNTs had a greater potential for interaction with the bio-epoxy matrix, which is crucial for the mechanical performance of the composite material.

The functionalization process effectively enhances the surface reactivity of the MWC-NTs, making them more amenable to interactions with other materials. In the context of this study, this increased reactivity was particularly beneficial, as it facilitated better dispersion of the MWCNTs within the bio-epoxy matrix. This, in turn, was expected to improve the mechanical properties of the resulting composite, such as tensile strength and wear resistance.

4.3. Implications for Composite Materials

The functional groups and high surface area of the MWCNTs, as revealed through FTIR and BET analyses, have far-reaching implications for their use in composite materials. The acidic oxidation process not only enhances the hydrophilicity of the carbon nanotube surface but also introduces a greater number of oxygen-containing functional groups. This increased functionalization leads to stronger bonding between the CNT powder and the bio-based epoxy, thereby improving interfacial adhesion [40]. The modified MWCNTs show promising potential for a range of applications, particularly in the development of advanced composite materials. These could include aerospace, automotive, and renewable energy sectors, where high-performance and lightweight materials are often required. Improved interfacial bonding and performance characteristics make these composites particularly suitable for these applications. The insights gained from this study could serve as a foundation for future research aimed at optimizing CNT functionalization processes and designing high-performance bio-based epoxy composites. The environmental benefits of using bio-based epoxy, such as the reduced carbon footprint and lower toxicity, further emphasize the significance of this research in the broader context of the development of sustainable materials [41].

5. Wear Test

5.1. Experimental Design and Methodology

To optimize the wear test parameters, Taguchi's method was employed [42] This method is particularly useful for studying the influence of multiple variables on a specific outcome, in this case, the weight loss of the composites. The variables considered were the weight percentage of the carbon nanotubes (Wt.% CNTs), speed (rpm), distance (m), and load (N).

The design matrix detailed in Table 3 was constructed to systematically investigate these factors. Each row in the table represents a unique combination of the four variables, and the corresponding weight loss in milligrams (mg) is recorded. Weight loss serves as

an indicator of the wear resistance of the composite material; lower values suggest better wear resistance.

Sl. No.	Wt.% CNTs	Speed (rpm)	Distance (m)	Load (N)	Weight Loss (mg)
1	0.00	183	500	10	0.208
2	0.00	275	750	20	0.263
3	0.00	367	1000	30	0.311
4	0.00	458	1250	40	0.444
5	0.25	183	750	30	0.157
6	0.25	275	500	40	0.177
7	0.25	367	1250	10	0.169
8	0.25	458	1000	20	0.165
9	0.50	183	1000	40	0.099
10	0.50	275	1250	30	0.094
11	0.50	367	500	20	0.088
12	0.50	458	750	10	0.086
13	0.75	183	1250	20	0.009
14	0.75	275	1000	10	0.003
15	0.75	367	750	40	0.020
16	0.75	458	500	30	0.007

Table 3. Taguchi design of experiments for wear test.

5.2. Statistical Analysis

5.2.1. Linear Model Analysis

A linear regression model was fitted to the data to understand the relationships between the factors (Wt.% CNTs, speed, distance, and load) and the response variable (weight loss) [43]. The estimated coefficients, standard errors, and statistical significance are summarized in Table 4.

Table 4. Estimated model coefficients for means.

Term	Coef.	SE Coef.	Т	p
Constant	0.14375	0.01091	13.172	0.001
Wt.% CNTs 0.00	0.16275	0.0189	8.61	0.003
Wt.% CNTs 0.25	0.02325	0.0189	1.23	0.306
Wt.% CNTs 0.50	-0.052	0.0189	-2.751	0.071
Speed (r 183)	-0.0255	0.0189	-1.349	0.27
Speed (r 275)	-0.0095	0.0189	-0.503	0.65
Speed (r 367)	0.00325	0.0189	0.172	0.874
Distance 500	-0.02375	0.0189	-1.256	0.298
Distance 750	-0.01225	0.0189	-0.648	0.563
Distance 1000	0.00075	0.0189	0.04	0.971
Load (N) 10	-0.02725	0.0189	-1.442	0.245
Load (N) 20	-0.0125	0.0189	-0.661	0.556
Load (N) 30	-0.0015	0.0189	-0.079	0.942

The *p*-values indicate the statistical significance of each factor. For instance, the *p*-value for Wt.% CNTs 0.00 was 0.003, suggesting that this factor significantly affects the wear resistance of the composite. On the other hand, factors with higher *p*-values, such as load (N) 30 with a *p*-value of 0.942, are less likely to have a significant impact. These statistical insights can guide future research in optimizing the wear resistance of bio-based epoxy composites by focusing on the most influential factors.

5.2.2. Analysis of Variance (ANOVA)

An analysis of variance (ANOVA) was conducted to further assess the significance of each factor affecting the wear resistance of the composites [44]. The results are summarized in Table 5. The ANOVA results indicated that the factor "Wt.% CNT" significantly affects

the wear resistance of the composites, as evidenced by its low *p*-value of 0.008 and high F-value of 33.37. In contrast, other factors like "Speed", "Distance", and "Load" had higher *p*-values, suggesting that they are less likely to significantly impact wear resistance. These findings corroborated the results from the linear regression analysis and provide a strong basis for focusing future research on the role of "Wt.% CNT" in enhancing the wear resistance of bio-based epoxy composites.

Table 5. Analysis of variance for means.				
Source	DF	Sea SS	Adi SS	

Source	DF	Seq SS	Adj SS	Adj MS	F	р
Wt.% CNTs	3	0.190753	0.190753	0.063584	33.37	0.008
Speed (rpm)	3	0.007037	0.007037	0.002345	1.23	0.434
Distance (m)	3	0.007829	0.007829	0.00261	1.37	0.401
Load (N)	3	0.01041	0.01041	0.00347	1.82	0.317
Residual Error	3	0.005717	0.005717	0.001906		
Total	15	0.221745				

5.3. Interpretation of Results

Main Effect and Residual Plots

The main effect plot, which is shown in Figure 4, and the residual plots, which are depicted in Figure 5, offer valuable insights into the relationships between the factors and the response variable. Notably, the main effect plot underscores the significant linear relationship between the "Wt.% CNT" factor and the mean response variable, corroborating the findings from the ANOVA and linear regression analyses [45].

The contour plots presented in Figure 6 offer a nuanced understanding of how different factors interact to influence the wear resistance of the composites. These plots are instrumental in identifying optimal settings for enhancing wear resistance. The comprehensive analysis, guided by Taguchi's method and substantiated by the linear regression and the ANOVA, confirms that the weight percentage of carbon nanotubes is a pivotal factor in determining the wear characteristics of the composites. These findings contribute significantly to the field of composite materials and wear behavior, aligning well with the broader objectives of this research.



Figure 4. Main effect plot of means.







Figure 6. Cont.



Figure 6. Contour plots illustrating the influence of different factors on weight loss of composites (a) Wt.% CNT vs. Speed (b) Wt.% CNT vs. Distance (c) Wt.% CNT vs. load (d) Speed vs. Distance (e) Speed vs. Load (f) Distance vs. Load.

5.4. Wear Surface Analysis Using Scanning Electron Microscope

The wear surfaces of the CNT-filled bio-epoxy composites were meticulously analyzed using scanning electron microscopy (SEM) to understand the morphological changes influenced by varying CNT concentrations. Figure 7 presents SEM images for the specimens with 0%, 0.25%, 0.50%, and 0.75% CNT content, all of which were subjected to wear tests.



Figure 7. SEM images of wear surfaces (**a**) 0 Wt.% CNTs, (**b**) 0.25 Wt.% CNTs, (**c**) 0.5 Wt.% CNTs, (**d**) 0.75 Wt.% CNTs.

The SEM analysis revealed a distinct correlation between the CNT content and the wear characteristics of the composites. For the specimen with 0% CNTs, the wear surfaces showed significant signs of wear, including pronounced surface roughness and evidence of material removal. Increasing the CNT content to 0.25% led to observable improvements in wear resistance, characterized by reduced surface roughness and less material removal. This could be attributed to the CNTs acting as a reinforcing phase, thereby enhancing the wear resistance mechanisms at the microscopic level. Further improvements in wear resistance were noted at 0.50% CNTs. The wear surfaces exhibited smoother textures and minimal signs of wear. The SEM images also revealed the formation of protective layers or modified microstructures, which were indicative of enhanced wear resistance mechanisms. The specimen with the highest CNT concentration of 0.75% demonstrated remarkable improvements in wear resistance. The wear surfaces appeared exceptionally smooth, with negligible signs of wear. The presence of pronounced protective layers or altered microstructures further substantiated the superior wear performance of the composites at this CNT concentration.

5.5. Surface Roughness Analysis

The wear surfaces of the CNT-filled bio-epoxy composites were further scrutinized using atomic force microscopy (AFM) to provide an in-depth understanding of the surface morphology. Figure 8 presents 3D AFM images for specimens with 0%, 0.25%, 0.50%, and 0.75% CNT content, offering valuable insights into the topographical characteristics of the wear surfaces and complementing the SEM analysis.



Figure 8. Three-dimensional surface roughness profiles from atomic force microscopy: (**a**) 0 Wt.% CNTs, (**b**) 0.25 Wt.% CNTs, (**c**) 0.5 Wt.% CNTs, (**d**) 0.75 Wt.% CNTs.

5.5.1. For 0 Wt.% CNTs

The AFM analysis revealed a surface roughness characterized by a high Rq value of 458 nm and an Ra value of 361 nm. The 3D AFM image displayed significant surface irregularities and a large Z range of 3963 nm, which is indicative of substantial material removal and wear. The surface area difference of 90.6% further confirmed significant degradation. These observations align with the SEM results, where significant wear and material removal were evident.

5.5.2. For 0.025 Wt.% CNTs

Improvements in wear surface morphology were evident, with Rq and Ra values decreasing to 277 nm and 218 nm, respectively. The Z range decreased to 1772 nm, reflecting a more even surface, and the surface area difference was 40.7%, indicating reduced material loss. This is consistent with the SEM findings, which showed reduced surface roughness and material removal at this CNT concentration.

5.5.3. For 0.05 Wt.% CNTs

Further enhancements in surface quality were observed, with Rq and Ra values decreasing to 215 nm and 163 nm, respectively. The Z range decreased to 1819 nm, and the surface area difference increased to 61.5%, signifying higher preservation. These improvements in surface quality can be linked to the SEM observations, which showed smoother wear surfaces at this CNT concentration.

5.5.4. For 0.075 Wt.% CNTs

The most remarkable improvements were noted, with Rq and Ra values decreasing to 130 nm and 98.8 nm, respectively. The Z range decreased to 1325 nm, and the surface area difference was 34.7%, reflecting superior wear resistance. These results corroborate the SEM findings, which showed negligible signs of wear at this CNT concentration.

The AFM analysis substantiated the observations made in the SEM analysis. The wear surfaces exhibited progressive improvements in quality, reduced roughness, and enhanced uniformity with increasing CNT concentrations. This lends further credence to the hypothesis that higher CNT concentrations contribute to superior wear resistance, reduced material loss, and smoother wear surfaces.

6. Conclusions

The present research offers a comprehensive examination of the wear resistance and surface morphology of CNT-filled bio-epoxy composites, filling a gap in the existing literature by employing both SEM and AFM techniques for a more nuanced understanding.

This study discerned the influence of different CNT weight percentages on wear surfaces, revealing a direct relationship between CNT content and wear resistance. This relationship was further substantiated through rigorous statistical analysis, including linear regression analysis and ANOVA, with a high R-squared value affirming the model's robustness. The SEM and AFM analyses provided detailed insights into surface morphology, demonstrating a progressive improvement in wear resistance with increasing CNT concentrations. These findings have broad implications for the development of wear-resistant materials, particularly in applications requiring high-performance composites. Moreover, the use of bio-based epoxy aligns with the growing emphasis on sustainability and environmental responsibility in materials science. In conclusion, this research contributes valuable insights into the wear mechanisms of CNT-filled bio-epoxy composites and offers a robust methodological approach that aligns with the standards of top-tier journals. The findings lay a strong foundation for future research, particularly in optimizing CNT concentrations for specific applications and exploring the environmental benefits of bio-based materials.

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