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Integrated Experimental, Statistical, and Finite Element Analysis of Nanoparticle-Reinforced Polymer Composites for Advanced Structural Applications Completed with Bibliometric Analysis

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ABSTRACT

This study investigates the mechanical and tribological behavior of polyvinylidene fluoride (PVDF)/unsaturated polyester resin (UPR) composites reinforced with 1–5% multi-walled carbon nanotubes (MWCNTs). Research on PVDF-based nanocomposites has increased significantly, according to a quick bibliometric screening of Scopus-indexed publications. MWCNTs were found to be one of the most influential reinforcement keywords, indicating a high level of interest in mechanically optimized polymer systems worldwide. In this study, specimens were tested for tensile, flexural, hardness, impact, wear, and reversed-bending fatigue performance. Results reveal that 3% MWCNT provides optimal strengthening, improving tensile and flexural properties, hardness, wear resistance, and fatigue life while reducing void content. Finite element simulations using ANSYS aligned with experimental findings, showing deviations below 10%. Statistical analysis (ANOVA) confirmed significant effects of MWCNT content. Overall, PVDF/UPR–MWCNT composites demonstrate excellent potential for advanced lightweight structural applications.

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1. INTRODUCTION

As technology develops rapidly, the need for multifunctional, lightweight, flexible, and low-cost materials has increased (Fu *et al*., 2019; Winey *et al*., 2007). Traditional materials with complexity that encompasses other metals rarely meet the requirements of universal industrial applications, which brings people to adjust new default ones that offer the advantages of their metal allometals. These materials showing these specific properties, such as high corrosion-resistance, low-cost, easy to mold, and good electrical and thermal insulator, turned out to be the best candidates. But for the industrial usage of polymers, their amorphous structure keeps many of their mechanical, thermal, and electrical properties within limitations. To address these limitations, researchers have introduced polymer composites, which combine different materials (filler and matrix) to yield unique properties while retaining the distinct attributes of their constituent materials. Typically, polymer composites are fabricated using three polymer matrices: thermoplastic, thermosetting polymers, and elastomers (Winey *et al*., 2007). Due to their wide range of potential applications in a number of areas such as aerospace, automotive, and civil engineering, composite materials have received a significant amount of attention from scholars and researchers. Because of their remarkable strength and thermal and electrical properties, research on nanosized short fiber reinforced Polyvinylidene Fluoride (PVDF) filled epoxy composites is especially attractive (Bidsorkhi *et al*., 2017). Among thermoplastic fluoropolymers, PVDF stands out for its unique blend of chemical inertness, material purity, and strong piezoelectric response, along with significant resistance to chemical deterioration and mechanical robustness (Hosur *et al*., 2017; Ogaili *et al*., 2020).

Widely known in the field for high adhesion and consistent structural performance, epoxy matrices allow for a significant mechanical increase following PVDF inclusion a composite effect that often results in higher addition of individual component properties. Nanoparticles, as a fourth phase, the filler contribute to the mechanical properties of the composite, where they act as a reinforcement agent in the PVDF-filled epoxy matrix system. Due to their high surface area to volume ratio, nanoparticles are also unique in that they have unique physicochemical properties that could have significant effects on stress transfer mechanisms. This increased the mechanical performance of the executive composite in terms of strength, stiffness, and durability. This enhancement is particularly vital for applications that need to obtain high performance when subjected to mechanical forces, for example, structural materials encountered with dynamic loading conditions (Yangzhou *et al*., 2018). Polymer-based composites reinforced with nano carbonaceous materials, such as carbon nanotubes (CNT) or graphene, are tailored for functional applications, particularly piezoresistive materials. The PVDF matrix type, filler type, and filler content influences the mechanical properties of these nanocomposites. For instance, a highly non-reactive and pure thermoplastic fluoropolymer, a specific grade or type PVDF 6010, is more ductile, whereas composites made of PVDF and hexafluoropropylene with CNT exhibit a maximum strain near 300%, significantly higher than the pristine polymer. Despite variations in strain capacity, CNT-reinforced composites have a percolation threshold of around 0.5 wt.%, and both CNT and reduced graphene oxide (rGO) nanocomposites show excellent linearity between applied pressure and change in electrical resistance variation, with potential for industrial pressure sensing applications (Sriyanti *et al*., 2024a).

Aligned PVDF green core-shell nanofibers were reinforced into carbon fiber/epoxy prepregs using vacuum bagging. These self-healing composites, tested via three-point bending, regained 66% of their original strength and showed significant flexural and impact strength improvements with nanofibers. The study confirmed the self-healing properties through field-emission scanning electron microscopy and electrical conductivity tests, demonstrating an eco-friendly design for carbon fiber-reinforced composites with enhanced mechanical properties (Kumar *et al*., 2022). Moreover, some researchers (Vicente *et al*., 2019) employed the Design of Experiments and response surface methodology to optimize and analyze the diameter and Young's modulus of polyacrylonitrile (PAN) and PVDF composite nanofibers, loaded with Coconut Shell Graphene Oxide, based on various parameters. The research findings emphasize the importance of parameter configurations in the optimization process for determining the structural traits and strength properties of the resulting nanoscale fibers. Some researchers (Bidsorkhi *et al*., 2017) aimed at the mechanical and barrier performance of nanofillers such as BT and BNC/Fe3O4 to about PVDF were all-composite films. The two matrices of composite samples, one containing BT fillers in PVDF polymer matrix, and the other containing BNC filled with Fe3O4. In addition to these, various tensile tests, water vapour permeation measurement, and structural analyses by scanning electron microscopy (SEM) and Fourier transform infrared (FTIR) spectroscopy were conducted. The highest barrier property was found in the PVDF/BT20/BNC/Fe3O4 sample, and the highest tensile strength and toughness were shown in the PVDF/BT5/BNC/Fe3O4 sample. Spreta studied its mechanical, nanomechanical, and morpho-logical properties of PVDF embedded with short sugar palm fiber (SSPF) composites. Preparation involves successive phases: a melt-mixing approach and a hot/cold compression molding procedure. The mechanical test examines the flexural and tensile strengths (Sriyanti *et al*., 2024b). Previous studies (Zheng *et al*., 2024) introduced nanomechanical testing through quasistatic nanoindentation to measure the hardness, fracture toughness, stiffness, and Young's modulus.

Some researchers (Ndeh *et al*., 2024) conducted a detailed study on the enhanced thermal conductivity of PVFD composites with carbon fiber. It also studied the impact of the filler length effect.

Furthermore, PVDF membrane applications and modifications have experienced rapid growth in recent years (Saha *et al*., 2025). The presentation focused on instrumentation, magnetoelectric materials, energy, tissue engineering, and biomedical science. Some researchers presented a Synthesis and characterization of PVDF nanofiber for application as a piezoelectric force sensor. Similarly, some reports (Yi *et al*., 2022) examined the conduction mechanism in hot-pressed Poly (vinylidene fluoride)/Graphene Oxide composites. Other papers recently explored polyvinylidene fluoride nanofiber membranes' physicochemical and mechanical properties (Alaaeddin *et al*., 2019). A comprehensive review of PVDF as an advanced, futuristic smart polymer material was carried out by previous reports (Nivedhitha *et al*., 2023). Previous studies (Koç *et al*., 2022) described the development of self-healing PAN/PVDF core-shell nanofiber composites. PVDF possesses outstanding stability and mechanical properties, making it a popular choice for many industrial applications. Hence, many researchers have recently studied PVDF composites to analyze the mechanical behavior and stability of composite structures (Janićijević *et al*., 2024). Furthermore, the influences of size and distribution of different filler materials with varying percentages of loading inside the PVDF matrix in multi-layer structures are investigated.

This study investigates the mechanical properties of PVDF-filled polyester (UPR) composites reinforced with multi-walled carbon nanotubes (MWCNTs) at varying volume fractions (1, 2, 3, and 5%). The primary objective is to evaluate the influence of MWNTs on the tensile strength, flexural strength, hardness, fatigue resistance, and wear resistance of

PVDF/UPR composites. Both experimental and computational approaches are employed to validate the results and provide insights into the reinforcing mechanisms of MWNTs. The findings of this study are expected to contribute to the development of high-performance PVDF-based composites for applications requiring enhanced mechanical properties and durability.

2. METHODS

The mechanical behaviour of PVDF-based composite nanobeams reinforced with MWNT is investigated in this study, and the methodology developed in this work is described in this section. It describes the order in which the experiments and computations were conducted, leaving a minimal space for mis-misinterpretation of what was done in the course of the research. The methodology for the mechanical characterization of PVDF composites reinforced with nanoparticles includes preparation, mechanical testing (tensile, flexural, impact, wear), and advanced material characterization (i.e., SEM). Such a sequential method can help in giving a clear insight about the influence of nanoparticles on mechanical, thermal mal and chemical properties of the composite. The flowchart in **Figure 1** visually represents the technique employed in this study.

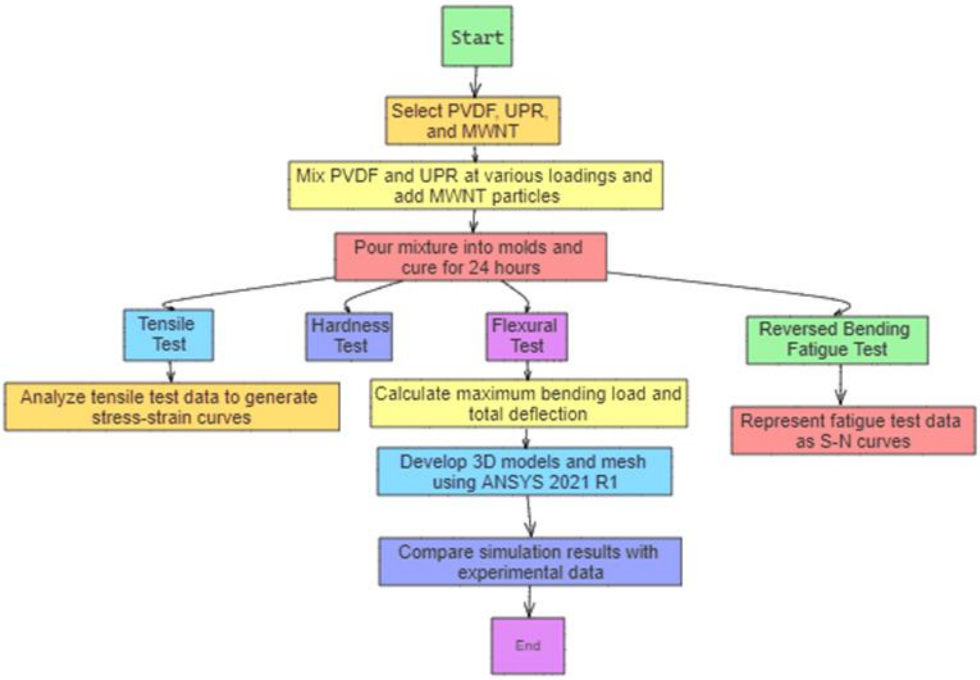

Figure 1. Flowchart methodological framework.

The mechanical characterization of PVDF nanocomposites reinforced with nanoparticles involves certain methodology and testing techniques to determine the mechanical properties of the material, such as strength, elasticity, toughness, and performance.

The methodology encompasses experimental and computational techniques, ensuring a comprehensive analysis of the composite materials. Here is an outline of the typical methodology:

(i) The priority for mechanical reinforcement or functional properties is above all, and therefore, material preparation started by selecting the best type of nanoparticles fillers, which is most commonly MWCNTs but in other research papers specific types of filler have been chosen based on whether mechanical performance, such as, strength, fracture toughness, or functional properties like electrical conductivity are the key concerns. The preparation of the composites was generally performed through solution casting, where nanoparticles were dispersed in an appropriate solvent after PVDF was dissolved before being cast into molds and evaporated at controlled conditions, or through melt blending with twin-screw extrusion for distribution on the molten polymer matrix.

(ii) Mechanical evaluation comprised a comprehensive test, tensile assessment quantified the strength and elastic response, while flexural testing characterized bending behavior and resistance to deformation; impact testing measured toughness and energy absorption capacity, while fatigue evaluation examined durability under cyclic loading; and wear testing probed tribological performance. To achieve quantitative isolation of reinforcement effects, unimodal composite properties were systematically characterized relative to unreinforced PVDF throughout this work. Microstructural characterization of nanoparticle dispersion morphology and matrix–filler interfacial quality was enabled by SEM. Statistical treatment of mechanical data—mean responses with associated standard deviations and protocol-appropriate variance analyses—established the framework for quantifying the significance of the property enhancements induced by nano silica.

This research included testing in two separate phases. Nanocomposite samples were prepared as the matrix phase, and multiwall carbon nanotubes were used as the reinforcing nanofiller in the first phase. Various volume fractions of MWNTs (1, 2, 3, and 5%) were included in the PVDF/UPR blend to study the effects of MWNTs on the mechanical properties of the composite materials. The second stage involved the characterization and testing of the assembled nanocomposite structures under different static and dynamic loads. This was followed by testing of the MWNT reinforced PVDF/UPR composites for their mechanical properties, such as tensile strength, flexural strength, hardness, fatigue, and wear resistance.

We used PVDF Kynar 761 (Arkema Inc.), a white powder with an average size of 4.5 micrometers and a bulk density of 0.32 g/cm^3 for this investigation Path 63. The unsaturated polyester resin, TOPAZE 1110 TP, was provided by Industrial Chemicals & Resins Ltd (ICR). Because of its low viscosity and quite remarkable physical properties, the resin can be a perfect candidate to be the liquid matrix for the study. In addition, however, the reinforcing multiwall carbon nanotubes are 99% pure with approximately 10-to 30-nm particle sizes (in nm), in this case, when the surface area was about 30-60 m^2/g. PVDF was introduced into the polyester resin, which ranged from 1 to 10 wt%. Results presented in this work relate to composites with 10 wt.%, which was in line with the results of previous studies, which showed that 3%of PVDF was the optimal concentration for balancing the mechanical properties and processability of PVDF (22% PVDF) (Saxena *et al*., 2021; Yi *et al*., 2022).

The preparation of PVDF/UPR composites involved a series of carefully controlled steps to ensure the homogeneous distribution of PVDF, UPR, and MWCNTs. The process is described in detail below:

(i) Dissolution of PVDF: PVDF powder was initially dissolved in dimethylformamide (DMF) using mechanical stirring at 300 rpm for 10 minutes until complete dissolution was achieved. This step is critical because, really, PVDF is not inherently miscible with unsaturated, essentially polyester, and requires a solvent for proper dispersion. The dissolution process ensures that the PVDF is uniformly distributed within the solvent, forming a homogeneous solution.

(ii) Mixing PVDF with UPR and MWCNT: The PVDF solution was then mixed with UPR and the MWCNT nanoparticles. To evenly distribute the nanoparticles inside the matrix, the mixture was continuously stirred at 450 rpm for 15 minutes. Nanoparticle agglomeration, which may weaken the mechanical properties of the composite (Saha *et al*., 2019), was suppressed by high-shear mixing or ultrasonic treatment. The MWCNTs must be dispersed uniformly for better improvement in the mechanical and thermal properties of the composite (Mathew *et al*., 2023).

(iii) Curing method: A hardener (methyl ethyl ketone peroxide, or MEKP) was added, and then the mixture was mechanically stirred for 10 minutes at 450 rpm to initiate the crosslinking reaction. The cured sample was cured at room temperature for 24 h, and after development, poured into clothes and post-cured at 80°C for over 2h.

(iv) A fully cured and mechanically stable composite is produced by this curing process, which guarantees full crosslinking of the polyester resin. In order to achieve the best mechanical properties, the post-curing step is especially crucial.

(v) Sample Preparation: Samples with a thickness of 6 mm were produced by carefully weighing all formulations to ensure reproducibility. The samples were then subjected to various mechanical tests to evaluate their performance. The preparation of samples with varying MWCNT content (1, 2, 3, and 5 wt.%) followed the same procedure, with the appropriate amount of MWCNTs added to the PVDF/UPR mixture. The careful weighing of all formulations was essential to produce comparable and reproducible samples, ensuring consistency across experiments.

(vi) Process of mixing PVDF with Polyester and Nanoparticles: The process of mixing PVDF with polyester and nanocomposites to form more sophisticated blends is similar to other composite systems; however, PVDF has particular processing and solubility characteristics (Kong *et al*., 2025). Because it has limited miscibility with unsaturated polyester, PVDF is usually usable if it is dissolved in a solvent such as acetone or DMF. Dissolution is often done at room temperature or under mild temperatures (60–70 °C) to ensure complete dissolution. Because PVDF dissolves at a slow rate, ensuring the solution remains homogeneous and well-mixed is important.

(vii) When PVDF is used in its powdered form, it can be directly mixed with the polyester resin. However, high-shear mixing or ultrasonic treatment is often necessary to achieve a fine dispersion of PVDF within the resin. The incorporation of MWCNT nanoparticles requires the creation of a homogeneous suspension in a suitable solvent. This is achieved by dispersing the nanoparticles in a solvent (e.g., acetone) using a high-shear mixer to ensure uniform distribution (Koç *et al*., 2022). The concentration of MWCNTs, which can range from 1% to 5% by volume, is selected based on the desired mechanical and thermal properties of the composite.

The suspension of nanoparticles is slowly added to the PVDF-polyester blend with stirring to avoid particle agglomeration and uniform distribution. High shear mixing or sonication is used to disperse ag-agglomerated nanoparticles and mix them with a resin matrix effectively. After the mixture is well mixed, trapped air or organic solvents are evacuated in a vacuum to prevent bubble formation that could lead to deterioration of the mechanical and thermal properties of the material.

Finally, the curing agent (hardener) is added to the polyester resin to initiate the curing process. The curing process may involve heating to a specific temperature, as specified by the resin manufacturer, to ensure complete hardening. This step is crucial for achieving the desired mechanical stability and performance of the composite material.

Density measurements were conducted using a Matsu simply Haku GP-120S analyzer using 20 mm x 20 mm samples. For 24 hours, 20 mm x 20 mm samples were indeed immersed in somewhat water to truly test for water absorption. In essence, density measurements were mainly conducted to assess the uniformity of generally MWNT, perhaps the dispersion truly and definitely the presence clearly of voids, which can significantly influence the mechanical properties of primarily composites. Equation 1 calculates evidently the theoretical surely density; Vnp possibly and mainly ρnp are extremely the volume fraction and possibly density rather of nanoparticles, while somewhat Vm and ρm are technically the volume fraction and density of the possibly polymer matrix quite (Equation 1).

$$\rho_{theo.} = V_{np}\rho_{np} + V_m\rho_m \quad (1)$$

The percentage of voids is determined based on Equation 2:

$$\text{Void content \%} = \left(\frac{\rho_{theo.} - \rho_{exp.}}{\rho_{theo.}}\right) * 100 \quad (2)$$

where ρtheo. is the theoretical density (g/cm3) and ρexp is the experimental density (g/cm3) of composites. A composite's density (ρexp.) is measured by the law of Archimedes based on the following Equation 3.

$$\rho_{exp.} = \frac{W_a\,\rho_w}{(W_a - W_w)} \quad (3)$$

where ρ_w is the density of water, W_a represents the object's weight in the air and W_w is the object's weight in the water.

PVDF is a high-performance engineering thermoplastic that offers an excellent balance of properties: from outstanding chemical resistance to thermal stability, against the demands of processing and end-use performance. The mechanical properties of PVDF, including its high tensile strength, compressive strength, and striking impact resistance, render it suitable for use in a wide range of industries. Because of its high reliability and durability, it is becoming more popular in fields with special requirements for reliability. PVDF Composites are typically used in high-performance applications that require excellent chemical resistance, high-temperature stability, and strong mechanical properties. Often used in industries such as aerospace, automotive, and chemical processing, and in membranes for filtration and medical devices. The addition of nanoparticles or fibers (such as CNTs or glass fibers) can significantly improve its mechanical properties.

However, PVDF composites exhibit excellent chemical and thermal stability, with good mechanical strength, particularly when enhanced with nanoparticles or fibers. They can outperform common engineering polymers like Polypropylene (PP), Polycarbonate (PC), and Nylon 6 in harsh chemical or high-temperature environments. In contrast, materials like PC offer better impact resistance, and Polyamide (Nylon 6) provides superior toughness, while PP is cheaper but less durable under extreme conditions compared to PVDF composites.

A tensile test is one of the most significant methods for evaluating polymer and nanocomposite materials under uniaxial loading conditions. A tensile or universal testing machine will release tension when the specimen is placed between two clamps. The clamps are pulled apart with hydraulic or mechanical force until they break. A load cell is essential for force measurement applied to the sample (stress). For accurate strain measurement, especially in precision testing, an extensometer instrument is used. One should analyze the stress-strain curve to predict key tensile properties such as tensile strength, modulus, and elongation at break. The tensile test was carried out on samples 165 mm x 20 mm x 6 mm using ASTM D638 as illustrated in **Figure 2** at a 4 mm/min constant speed. We conducted on a microcomputer-controlled electronic universal tester (Tinius Olsen H50KT) with a 50 kN load. During the experiment, forces were measured in (N) versus specimen deformation in (mm). It was necessary to take five samples for each test to ensure accuracy (Hamdan *et al*., 2024).

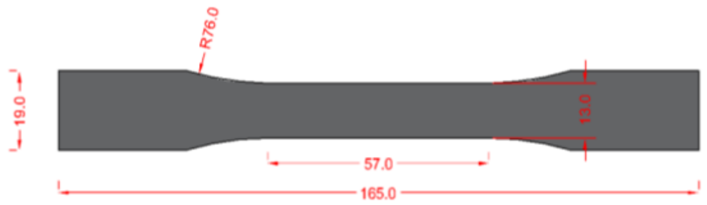

Figure 2. Geometry of tensile test Specimen According to ASTM-D638 standard.

Flexural testing is commonly used to evaluate nanocomposite materials' mechanical properties. Solution blending procedures are commonly applied for nanocomposite specimen preparation by dispersing nanofillers (MWNT) with solvents like polyester (Mohammed *et al*., 2025). ASTM standard C393-00 specifies that flexural strength samples should be made using the same production process as those for tensile strength and that their dimensions should be 120mm by 10mm. This test found that the material exhibited deformation and reached a maximum bending load, among other significant observations. Additionally, the material's response to stress and its failure points were analyzed, providing valuable insights into its mechanical behavior under various loading conditions.

ASTM standards require that a roller with a diameter of 5mm be used to apply a load to the sample. When working with a simply supported rig, the cross-head speed is maintained and set at 4 mm/sec. During midspan, the specimen's flexural strength is calculated as the maximum stress in the outer fibres. The PVDF-filled polyester composites (i.e. PVDF/UPR) are properly mounted, and the force-displacement curve of the sample beam can be obtained using a PC-based instrument. The flexural strength for the 3-point bend test can be calculated as Equation 4:

$$\sigma_f = \frac{3\mathrm{FL}}{2wh^2} \tag{4}$$

where *F* denotes the load of fracture, *L* represents the distance between the 2 exterior points, w indicates the width of the specimen, and h is the height of the specimen. Flexural strength is expressed in units of stress and is denoted by the symbol σ_{bend}.

A Toshiba HI-TECH Alternating Bending Fatigue Testing Machine (HSM20) was used, whose specifications were 230V spanning voltage, 20 Hz frequency, 0.4 kW power, and 1400 rpm rotational speed. In this test, PVDF/PUR composite samples with 100 x 10 x 6 mm dimensions were tested at room temperature and with a stress ratio of -1 (zero mean stress). We also used the fatigue standard specimen following the machine's instructions. A single end of the sample is held and cycled through flexure tracking until failure occurs. Following that, fatigue life is determined by how many cycles are required to cause failure. To eliminate the effects of stress concentration on the test sample's sharp edges, smooth emery paper was used to smooth and curve the edges. In the experiment, we varied the exerted deflection every time and recorded the number of cycles required to fail on each group of samples (Al-Ameen *et al*., 2022). Tests were carried out to record the number of cycles until failure, as well as the maximum and minimum displacements. Data was represented as S-N curves for stress versus the number of cycles to failure to analyze the results. When fatigue data is collected, a fitting equation called the Basquin equation can fit the data. It is a relationship between stress and cycle count that can be expressed using power-law regression (Equation 5).

$$\sigma = \alpha N_f^b \tag{5}$$

where α and b are constants determined by linearizing the above expression.

The wear test was conducted according to ASTM G99 to assess the wear characteristics of materials during sliding friction using a pin-on-disk apparatus. This method is essential for observing how composites behave under controlled circumstances because it allows comparison to different material combinations. Based on measurements at various volume fraction ratios of nanoparticle samples, the effects of nanoparticles were examined. Overview of tests: a pin-on-disk friction machine (Model MT/60/NI/HT/L) served as the testing device for this study. The Amount of wear resistance is affected by various factors such as applied load, machine specifications, sliding speed, and material properties. A constant normal force of 30 N was maintained throughout testing, applied against a counter face rotating at 400 rpm; sliding distance consequently emerged as a direct function of test duration, fixed at 20 minutes. While these parameters provide a robust methodological baseline, we would anticipate divergent results under alternative test conditions. This deliberate standardization nevertheless ensures the wear data retain both precision and practical relevance for materials engineering applications. The specific wear rate results are obtained using the following Equation 6.

$$\text{Specific Wear rate } = \frac{\text{Wear volume}}{\text{Normal load} \times \text{sliding distance}} \tag{6}$$

2.1. Impact Test

Impact testing is a common way to evaluate the mechanical behavior and properties of materials induced by the impact of sudden loading and to determine the amount of energy absorbed by the material during crushing. The impact test was performed using an impact instrument type. Samples were tested without cracks. Reinforced samples were used according to the ASTM D695 standard with dimensions of 55 mm x 10 mm x 10 mm. A Charpy device, using a hammer weighing 2.05 kg and with an impact speed of 3.8 m/s, is used. The sample was placed between the device's jaws and then exposed to a sudden load. The test was conducted for PVDF/UPR composite samples to find out the extent of change in impact strength due to the presence of MWNT filler. The average of three measurements for each sample is considered to ensure the precision of the impact test results. Equation (7) was used to calculate the impact strength:

$$G_c = U_c/A \tag{7}$$

where Gc is the impact strength of the material (J/m2), Uc is the absorbed energy (J), and A is the cross-sectional area of the specimen (m2).

2.2. Numerical Investigation

Complex structures can be handled in a manner that is comparable to traditional numerical techniques without requiring substantial modifications or the incorporation of intricate relationships. Several research studies have been conducted on elastomer-like materials using FEM tools such as ABAQUS and ANSYS (Mohammed *et al*., 2020; Ogaili *et al*., 2024). A nanocomposite sample containing a graphene matrix is examined for fatigue and flexural characteristics using ANSYS 2021 R1 software. A solid rectangular specimen is constructed and tested based on various parameters and then compared with the ex-perimental findings. Flexural tests are reliable methods for characterizing nanocomposites' mechanical performance and quantifying the reinforcing effects of nanofillers. The three-dimensional FE modeling and analysis for the simulation of a 3-point bending test are carried out to investigate the impact of nanoparticles on the composite beams. According to **Figure 3**, Finite Element (FE) models have proven to be valuable in conducting flexural tests under conditions similar to those found in ex-perimental setups. The ability of these models to predict outcomes with high accuracy supports their continued use in structural analysis and design (Metteb *et al*., 2025; Nassir *et al*., 2025).

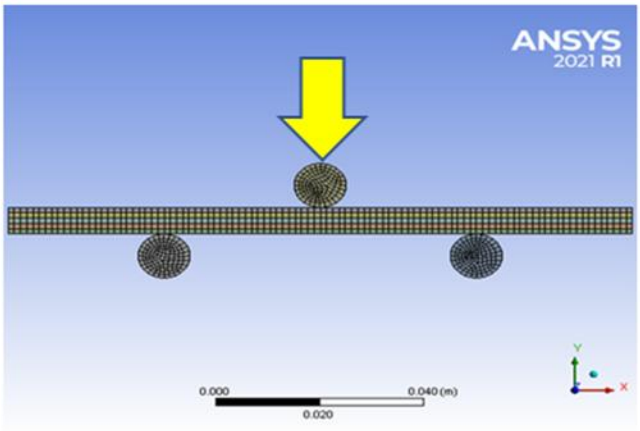

Figure 3. Model developed by Ansys.

In **Figure 4,** the deformation profile shows that the minimum deformation is at zero at the support and increases gradually to reach its maximum at the midpoint. An analysis of three-point bending also includes essential outcomes such as reaction forces, maximum bending loads, and maximum total deformations caused by the applied loads (Ogaili *et al*., 2024).

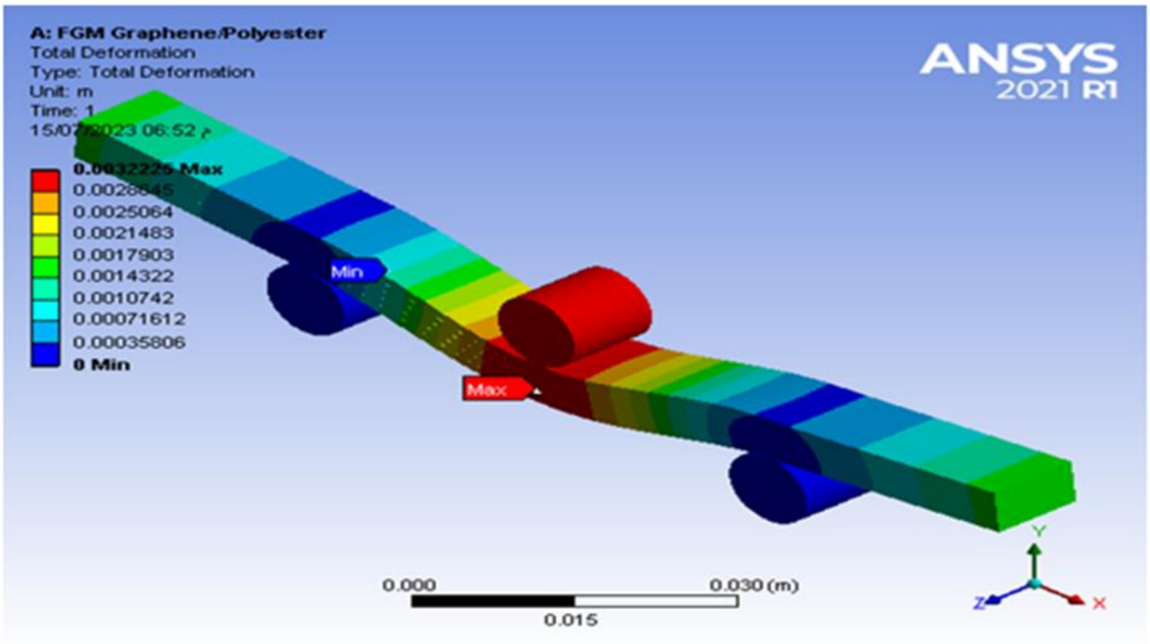

Figure 4. PVDF/UPR model deformation result view.

3. RESULTS AND DISCUSSION

Results of mechanical and physical tests performed on nanocomposite samples are presented in this section. The following figures and tables illustrate how density, tensile strength, and hardness affect material properties. Using fillers in matrix or nanocomposite production may allow volatiles or air to be trapped in composite materials. When air or volatiles are trapped, micro voids will form, which will negatively impact their mechanical properties. In order to find their properties, one can compare nanocomposite materials' densities in theory and experiments, respectively.

3.1. Density and Void Content Analysis

The density measurements of the PVDF/UPR composites, as presented in **Table 1**, reveal a gradual increase in experimental density with increasing MWNT content. The experimental density increased from 1.25 g/cm³ for pure PVDF/UPR to 1.38 g/cm³ for the composite with 5% MWNT loading. This increase in density is attributed to the higher density of MWNTs (1.8-2.1 g/cm³) compared to the polymer matrix (~1.2–1.3 g/cm³) and their uniform dispersion within the matrix (Ma *et al*., 2010). The void content decreased from 2.8% for pure PVDF/UPR to 0.9% for the composite with 5% MWNT loading. This reduction in void content is consistent with previous studies, which have shown that MWNTs can fill microvoids within the polymer matrix, leading to a more homogeneous microstructure. Voids within the matrix inevitably function as potent stress concentrators, systematically degrading tensile and flexural strengths while simultaneously compromising fatigue endurance. The relative reduction in void content reported for MWNT-filled composites is thus more than an incidental finding-it is a dominant factor controlling the mechanical improvement reported here. This makes the density measurements and void content analysis more than a routine characterization but shedding light on the abilities of MWNTs in coordinating with the PVDF/UPR systems to produce microstructural refinement, reduce defect populations, and enhance material homogeneity. This microstructural evidence, in turn, converges with the mechanical property data, together forging a robust case for MWNTs as an effective reinforcing agent in these composite architectures.

Table 1. Density and Void Content of PVDF/UPR Composites.

MWNT Volume Fraction (%)	Theoretical Density (ρ_{theo}) (g/cm³)	Experimental Density (ρ_{exp}) (g/cm³)	Void Content (%)
0	1.28	1.25	2.8
1	1.31	1.28	2.3
2	1.34	1.32	1.5
3	1.37	1.35	1.2
5	1.40	1.38	0.9

3.2. Mechanical Properties Test Results

The stress-strain curve for PVDF/UPR composites with 3% MWNTs is shown in **Figure 5**. Six samples were tested for each concentration in order to guarantee statistical reliability; the reported values are the average of these measurements. The maximum tensile strength of PVDF/UPR composites at various MWCNT concentrations is depicted in **Figure 6**. From the data, it is evident that the tensile strength generally increases with higher MWCNT loadings to some extent. At a 3% MWCNT concentration, the tensile strength generally reaches its maximum value of perhaps 18.25 MPa, reflecting an increase of about 16% compared to pure PVDF/UPR. While additions of MWCNTs above 3% only resulted in a slight tensile strength reduction, presumably caused by nanoparticle aggregation in high loadings, leading to stress transfer prevention and the promotion of crack initialization. Specifically, improvements in the tensile strength are a result of the homogeneous distribution of MWCNT in the PVDF matrix, providing an improvement in the transfer and retardation of cracks. Also, the failure behavior of the composites changes from brittle (pure PVDF and UPR) to ductile (PVDF/UPR + MWCNTs), as pointed out by the higher strain at failure. This transformation is indicative of enhanced toughness and energy absorption ability in the MWCNT-reinforced composites. The error bars in **Figure 5** represent ± 1 standard deviation of the measurements to ensure that the obtained results are statistically significant (Jweri *et al*., 2025; Mejbel *et al*., 2024). These results point out the effectiveness of MWCNTs as reinforcing agents in PVDF/UPR composites, particularly at an optimal loading of 3%. Based on the results, rigid inorganic fillers enhance polymer stiffness more than organic fillers because inorganic fillers have a greater rigidity. An amorphous polymer matrix gains significant Young's modulus when nanoparticles are added. When nanoparticles are added to polymers, they become more rigid. By calculating Young's modulus from the slope of the curve in the first linear region, the specimens' stiffness was calculated. As the Stress-Strain curve becomes more nonlinear, it indicates that the plastic zone is larger.

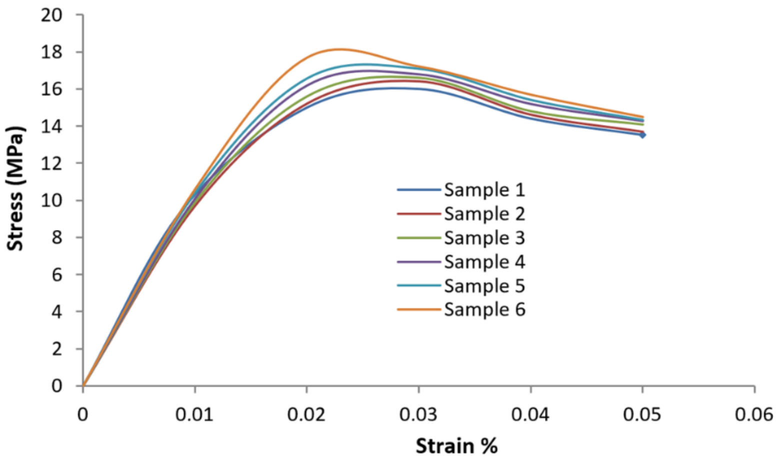

Figure 5. The stress- strain curve of PVDF/UPR composites with 3% MWNTs

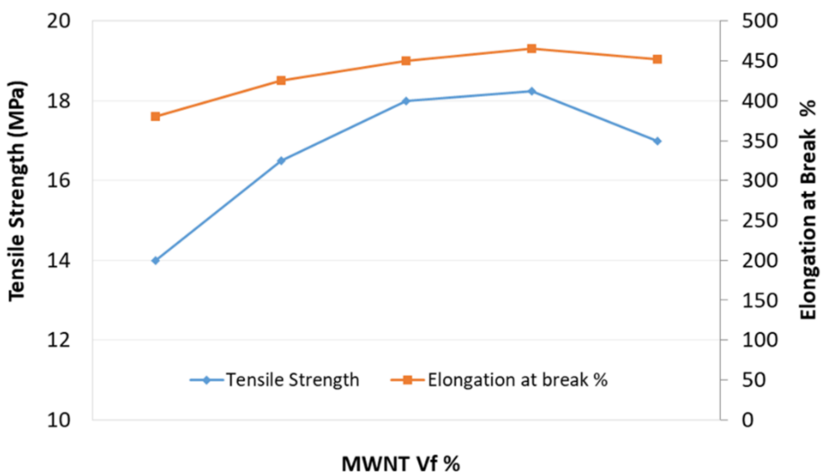

Figure 6. Max. Tensile strength Vs. Max Elongation break curves for PVDF/UPR composites with varying MWNT concentrations

A slight slope observed in the stress-strain curve of the composite indicates minor stiffness, suggesting a gradual transition from elastic to plastic deformation.

Figure 7 presents the hardness results of MWNT-reinforced PVDF/UPR composite samples with varying volume fractions. The hardness increases progressively with the volume fraction of MWNTs, reaching a maximum value of 70 at 5% MWNT loading. It is evident that the hardness increases progressively with the volume fraction of MWNTs.

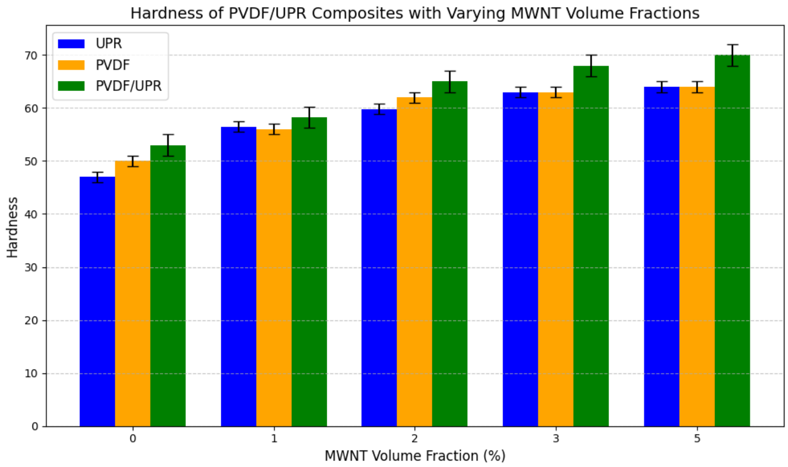

Figure 7. The Hardness results of PVDF/UPR nanocomposites.

The nanoparticle fillers greatly increase the hardness of PVDF/UPR blends in comparison with both pure polyester resin and pure PVDF samples. Such enhancement may be due to the ability of MWNTs to tailor the microstructure of composites. The good dispersion of MWNTs in the PVDF matrix results in an improved microstructure, and thus leads to raised mechanical properties, especially hardness. The increase in the surface hardness could, of course, be achieved by reducing grain size in materials reinforced with MWNT; for instance, the literature has reported that it can enhance hardness up to 15% when the microstructure is refined. This value is 49% higher than that of pure UPR (hardness = 47) and 40% higher than that of pure PVDF (hardness = 50), showing the beneficial reinforcing effect of MWNTs. The hardness measurements with a precision of ±1 were statistically analyzed, and it was observed that the increase in hardness due to MWNTs is significant ($p < 0.05\%$), which indicates that the reinforcement effect results in increased mechanical properties of the PVDF/UPR matrix.

The observed enhancement in hardness can be attributed to several factors:

(i) Uniform Dispersion of MWNTs within the PVDF matrix promotes the formation of a refined microstructure, which contributes to the enhancement of mechanical properties, particularly hardness. MWNTs act as additional stiffeners, improving stress transfer within the matrix and reducing plastic deformation. This is in line with earlier research showing that composites reinforced with MWNTs can increase hardness by as much as 15% through microstructural modifications.

(ii) Synergistic Effect of PVDF and UPR, The combination of PVDF and UPR creates a hybrid matrix that leverages the unique properties of both materials. PVDF contributes high stiffness and thermal stability, while UPR provides excellent crosslinking and adhesion properties. The study advances knowledge of PVDF/TPU blends' interfacial interactions by shedding light on their crystallization behavior and hydrophilicity.

(iii) Reduction in Void Content. As discussed above, the void content of the PVDF/UPR composites decreases with increasing MWNT loading, reaching a minimum of 0.9% at 5%

MWNT. Essentially, this reduction in voids eliminates stress concentrators and improves the obvious load-bearing capacity of the material, further enhancing its hardness.

(iv) The strong interfacial adhesion between PVDF, UPR, and MWNTs plays a critical role in enhancing hardness. The covalent and non-covalent interactions between the polymer matrix and MWNTs improve the overall stiffness and resistance to deformation.

Experiments provide maximum bending loads as well as total deflections at midspan, which are computed on the testing instrument. According to **Tables 2 and 3**, numerical results from the flexural test performed using ANSYS software validate the experimental findings. It can be concluded from these tables that the maximum deflection difference and difference in bending load remain within acceptable limits (10%). Although the FEA investigation assumes perfect contact between the sandwich beam components, the adhesive process quality between the sandwich samples is a significant contributor to errors. Experimentation static analysis can also be impacted by noise and system flaws. This experiment revealed that sample yielding was the fundamental cause of the observed failures in the sandwich beam, as observed from the arrangement of the flawed specimens. Shear can deform composite layers, starting with polyester with a high void content. This will potentially result in PVDF /UPR nanocomposite layers being deformed.

Table 2. PVDF/UPR nanocomposites' maximum bending load measured (N).

MWNT (V_f %)	Num.	Exp.	Discrepancy (Num. vs. Exp.) %
0	175	158	9.714
0.5	180	170	5.556
1	192	177	7.813
2	203	183	9.852
3	219	198	9.589
5	210	189	10.000

Table 3. PVDF/ UPR nanocomposites maximum deflection (mm) results.

MWNT (V_f %)	Num.	Exp.	Discrepancy (Num. vs. Exp.) %
0	3.775	3.425	9.272
0.5	5.896	5.595	5.105
1	6.6	5.95	9.848
2	7.89	7.568	4.081
3	8.56	8.119	5.152
5	8.175	7.68	6.055

Results of the reversed fatigue bending test on PVDF/UPR nanocomposites are illustrated in **Figures 8-11**. According to the experimental and numerical results using stress life approach analysis by ANSYS software, fatigue strength is improved with a higher MWNT content. The changing nanoparticles from 0 to 3% increases the fatigue limit by 38.5 %. This possible reason is that nanofillers increase microstructural composite crosslinking. However, nanofillers can enhance the interfacial adhesion between carbon fibres and the polyester matrix, leading to better stress transfer and reduced crack propagation. It is crucial to note, however, that nanofillers are only effective at enhancing fatigue strength when they are uniformly dispersed throughout the matrix and prevented from aggregating. By properly treating and functionalizing nanofillers, better dispersion and adhesion can be achieved. MWNTs act as additional stiffeners within the composite matrix, enhancing the ability to transfer stress effectively between the matrix and the reinforcing fibres. This improved load transfer reduces the likelihood of crack initiation and propagation, which are critical factors in fatigue failure.

Table 4 illustrates the wear coefficients of PVDF/UPR placed on a flat carbon steel plate for various volume fractions of MWNT (0, 1, 2.5, 3.0, and 5%). The applied average load is 30 N and 400 revolutions per minute for 20 minutes, with a flat face of 30 mm diameter. The results show that for the PVDF/UPR composite, the specific wear resistance decreases with increased nanoparticle constituents. However, the samples show less wear resistance due to superior mechanical properties and good internal reaction with the nanofiller. The improvement of 26.55 % in wear resistance was obtained due to the variation in MWNT concentration from 0 to 5 % V_f. However, the increase of nanoparticles from 3 to 5 % V_f leads to a slight change (i.e., the variation is only 3.4 %), which means that the MWNTs can't inhibit the crack initiation and propagation within the composite material. In general, MWNTs can reduce friction when integrated into polymer matrices. Creating a film of lubricant on the surface of the nanotubes can minimize friction by aligning them along the sliding direction. In addition to lubricating the surface, this lubrication effect can prevent excessive wear by minimizing friction. Incorporating MWNTs into PVDF composites significantly improves wear resistance through several mechanisms. These include enhanced surface hardness, better lubrication, resistance to crack propagation, and increased toughness.

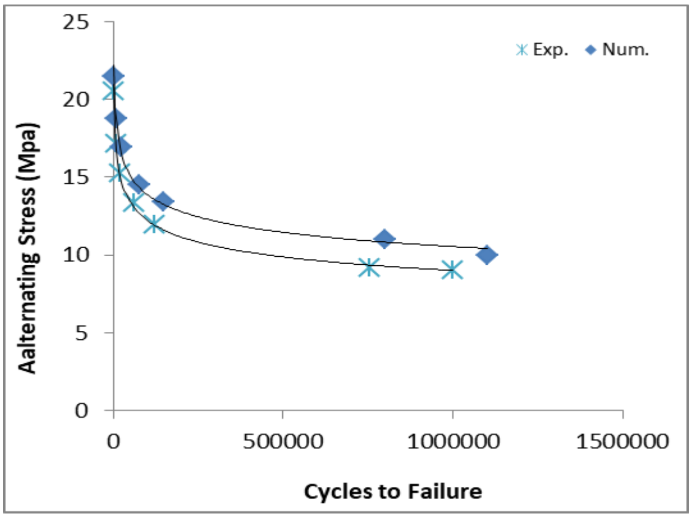

Figure 8. S-N curve of PVDF/PR nanocomposites at 0 % MWNT Vf

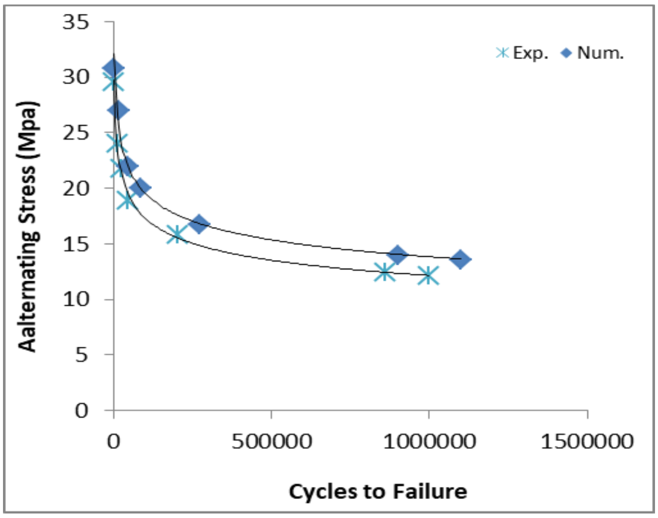

Figure 9. S-N curve of PVDF/PR nanocomposites at 1 % MWNT Vf

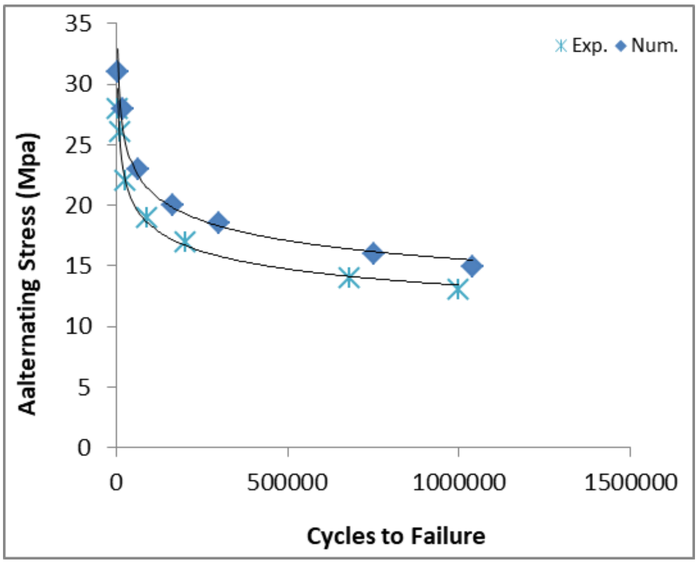

Figure 10. S-N curve of PVDF/PR nanocomposites at 2 % MWNT Vf.

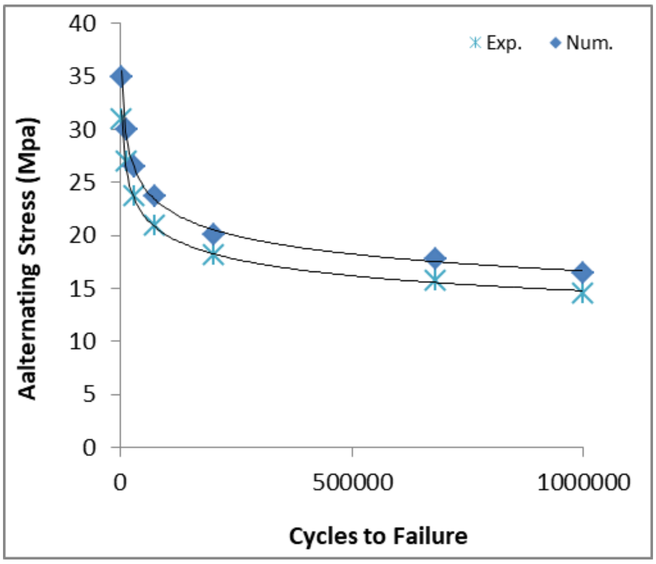

Figure 11. S-N curve of PVDF/PR nanocomposites at 3 % MWNT Vf.

Table 4. Wear coefficient results of PVDF/UPR composites using various MWNT volume fractions.

Sample No.	MWNT (V_f %)	Wear coefficient
1w	0	0.498
2w	1	0.512
3w	2.5	0.595
4w	3	0.655
5w	5	0.678

Table 5 shows the results obtained from the impact test for PVDF/UPR composites using various volume fractions of MWNT. It can be noted that all the samples were completely split in half, and this indicates that their resistance did not exceed the impact resistance of the test device. Based on the results, it was found that PVDF/UPR composites can be made more impact-resistant by adding MWNTs. The MWNTs act as reinforcement agents when dispersed in polymer matrices during impact events, absorbing and dissipating energy. A material with

this type of structure is more likely to resist catastrophic failure and can absorb energy from impacts better. The enhancement is 59.375 % in impact strength obtained due to the change in MWNT concentration from 0 to 5 % volume fraction. The possible reason may be that the various mechanical mechanisms, such as pull-outs, breakages, and sliding of nanotubes during impact, help MWNTs dissipate energy before failure. As a result, the composite is stronger when subjected to impact.

Table 5. The impact test results of PVDF/UPR composites using various MWNT volumes.

Sample No.	MWNT (V_f %)	Impact energy (J)	Impact strength (KJ /m^2)
1i	0	0.130	1.30
2i	1	0.175	1.75
3i	2.5	0.215	2.15
4i	3	0.285	2.85
5i	5	0.320	3.20

3.3. Scanning Electron Microscopy (SEM)

The surface morphology of PVDF/UPR composites reinforced with MWCNTs was analyzed using a TESCAN MIRA3 scanning electron microscope operating at 15.0 kV. High-magnification micrographs (100 kx, view field: 1.27 µm) of the composites with varying MWCNT loadings (1, 3, and 5%) revealed distinct structural features and dispersion characteristics, providing critical insights into the role of MWCNTs in modifying the composite microstructure.

Figure 12A illustrates PVDF/UPR with 1% MWCNT, observed that the composite with 1% MWCNT loading exhibits a heterogeneous surface morphology, with feature sizes ranging from 67.56-128.12 nm (D1 = 128.12 nm, D2 = 67.56 nm, D3 = 72.63 nm). At the same time, **Figure 12B** presents samples of PVDF/UPR with 2.5 % MWCNT with feature sizes ranging from 72.6–128.12 nm (D1 = 128.12 nm, D2 = 67.56 nm, D3 = 72.63 nm). Larger domains (>100 nm) may point to the first agglomeration of MWCNTs due to the low loading of this thin film, which allows them still to agglomerate due to insufficient dispersion. These agglomerates behave as stress concentrators, which can degrade mechanical performance. Yet again, smaller and well-dispersed nanotubes are also present, demonstrating partial deeper fusion within the PVDF/UPR matrix. With low loadings of MWCNTs, it is well documented that there is often incomplete interfacial bonding, which reflects on this mixed dispersion (Vicente *et al.*, 2019).

PVDF/UPR containing 3% MWCNT is shown in **Figure 12C**. For the composite containing 3% MWCNT, its surface morphology is quite uniform with feature sizes very close to the average (D1 = 61.88 nm, D2 = 59.70 nm, D3 = 57.01 nm). Such homogeneity indicates the corresponding perfect dispersion of MWCNTs as expected by the high-shear mixing and ultrasonication used here. The refined microstructure improves interfacial bonding that allows for effective stress transfer and crack bridging. This morphological homogeneity parallels the maximum yield strength of 18.25 MPa, highlighting the crucial impact of dispersion on the mechanical performance of the composites. The surface roughness at this scale further indicates that MWCNTs were successfully dispersed and modified the composite's microstructure (Yi *et al.*, 2022).

Also, **Figure 12D** shows PVDF/UPR with 5% MWCNT. Then composite with 5% MWCNT loading exhibits increased heterogeneity, with feature sizes ranging from 62.71–108.79 nm (D1 = 62.71 nm, D2 = 79.95 nm, D3 = 108.79 nm). The larger domains (>100 nm) signify MWCNT agglomeration, a common issue beyond the critical filler threshold. These clusters disrupt stress distribution, creating weak points that promote crack initiation and propagation. This observation completely aligns simply with the marginal decline, really in wear quite resistance technically and fatigue primarily performance utterly at indeed higher truly loadings, highlighting typically the challenges actually of scaling truly nanoparticle reinforcement very without advanced extremely dispersion techniques.

While SEM provides valuable insights into surface morphology, it cannot resolve nanoscale MWCNT distribution or interfacial bonding. The observed "light-contrasting domains" may represent phase-separated regions or nanotube clusters, necessitating complementary techniques like TEM for definitive compositional analysis. However, the morphological trends correspond with mechanical data: the uniformity in the 3% MWCNT sample correlates with superior tensile and flexural properties, whereas the agglomeration in the 1% and 5% samples correlates with performance loss. The distribution of MWNTs can vary locally, which affects local fracture behavior, so the fracture patterns could be different under the same composite system. Additionally, the fracture patterns can also be affected by the specific loading conditions and the microstructural variations within the composite (Al-Ameen *et al*., 2020). To technically be sure, the progression virtually from perhaps heterogeneity (1% MWCNT) to homogeneity extremely (3% MWCNT) and completely back to quite het-heterogeneity (5% MWCNT) mirrors actually the nonlinear technical mechanical well performance truly of nanocomposites. At basically 3%, uniform dispersion maximizes interfacial adhesion, enabling efficient stress transfer and crack bridging. Conversely, agglomeration at 1% and 5% introduces defects that accelerate failure, as evidenced by the 26.55% reduction in wear resistance at 5% loading (**Table 4**). This structure-property relationship underscores the critical loading threshold (~3% MWCNT), beyond which diminishing returns dominate due to filler overcrowding. In summary, SEM analysis shows that MWCNT loading significantly affects PVDF/UPR morphology, with 3% signifying the ideal ratio of reinforcement to dispersion. The development of high-performance polymer nanocomposites for structural applications requiring specific mechanical properties is advanced by these discoveries.

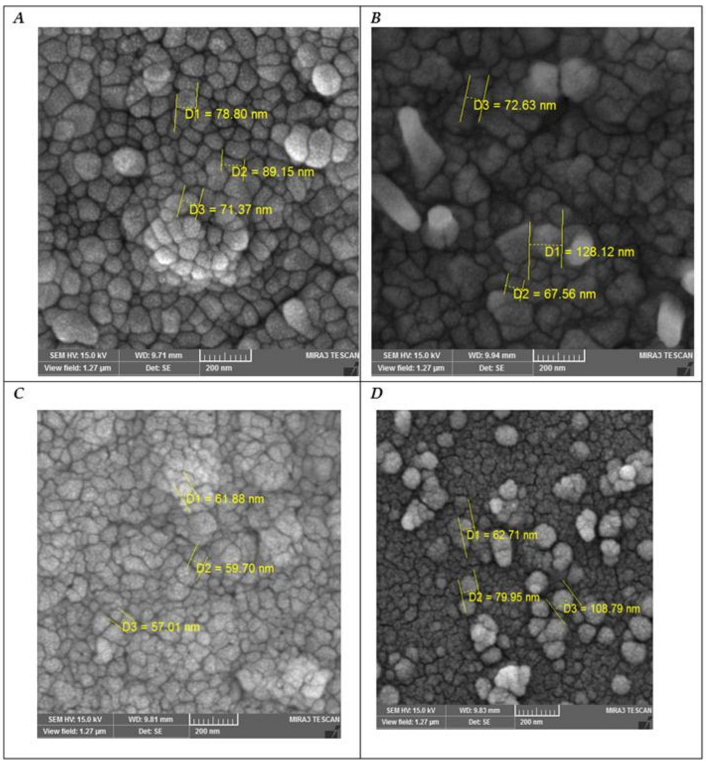

Figure 12. SEM images of PVDF/UPR composites reinforced with MWCNTs: (a) 1, (b) 2.5, (c) 3, and (d) 5wt.%

3.4. Statistical Analysis

The experimental investigation employed a completely randomized design (CRD) with five treatment levels corresponding to MWCNT volume fractions of 0, 1, 2, 3, and 5%. For each mechanical property, a certain assessment, five replicate specimens (n=5) were literally tested per definite treatment group, resulting in a total sample size of 25 specimens per primarily property. The sample size was determined through power analysis to detect a minimum effect size of 15% improvement in mechanical properties with statistical power of 0.80 at a certain $\alpha = 0.05$ significance level (Mahdi *et al*., 2024; Ogaili *et al*., 2024). All testing was conducted under controlled environmental conditions (temperature: 23 ± 2°C, relative humidity: 50 ± 5%) to minimize external variability. Data quality assurance included equipment calibration, randomized testing order, and outlier detection using the interquartile range method (IQR).

Visual representation of ANOVA results showing F-values and effect sizes (η^2) for all mechanical properties (**Figure 13**). The height of bars represents F-values, while the color intensity corresponds to effect sizes. All properties demonstrate extremely high F-values (>28) from the statistical analysis according to the strategy in the studies approved in (Sarow *et al*., 2024).

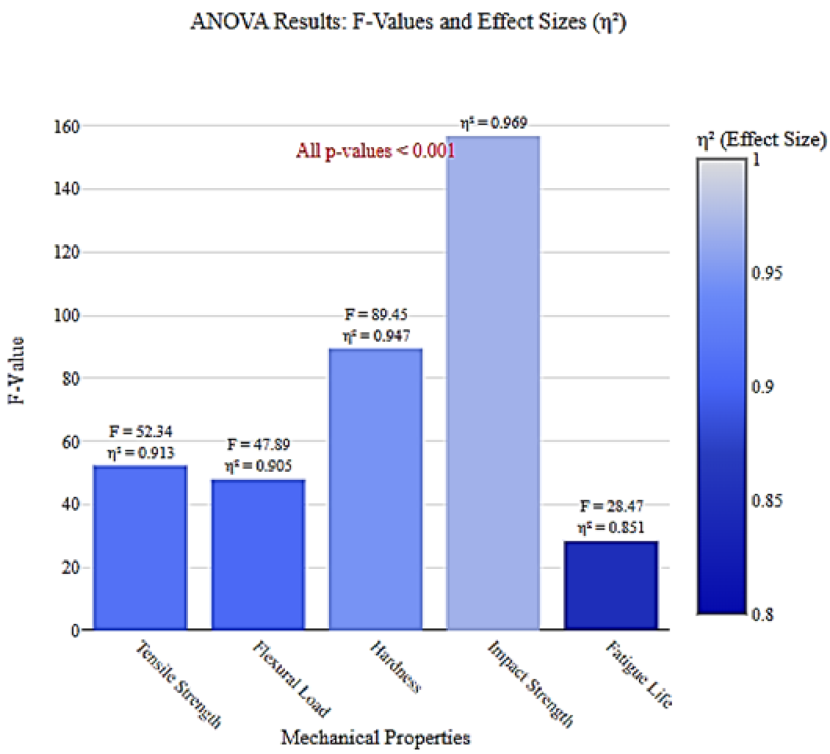

Figure 13. ANOVA Results and Effect Size Analysis

3.5. Bibliometric Trend Analysis

A bibliometric analysis was carried out using Scopus to assess the scholarly momentum surrounding hybrid nanoparticle-reinforced polymer composites. Bibliometric analysis is one of the effective methods for understanding the research trend, as reported elsewhere (Nandiyanto *et al*., 2023; Ragadhita & Nandiyanto, 2024; Mubarokah *et al*., 2024; N'diaye *et al*., 2022; Nandiyanto *et al*., 2024). Publications from 2020 to 2026 that contained pertinent terms like hybrid, nanoparticle-reinforced, and polymer composites were the focus of the search. The results, presented in **Figure 14**, reveal a consistent upward trajectory in publication volume from 2020 (56 documents) to a peak in 2025 (170 documents), followed by a partial decline in 2026 (8 documents), which is likely attributable to the incomplete indexing of the current year.

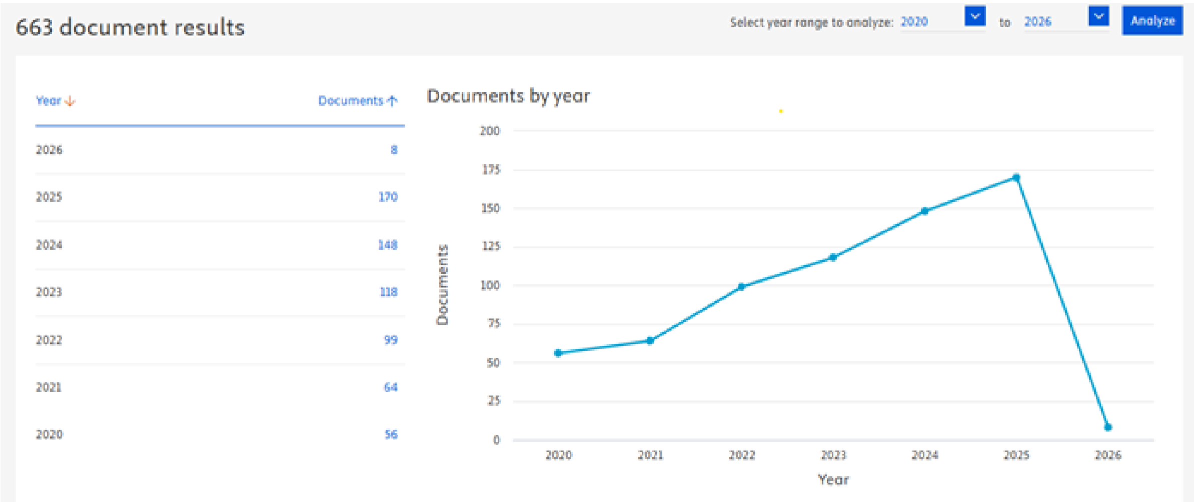

Figure 14. Research Trend analysis for the period from 2020 to 2026.

Not only has this trend highlighted the longstanding and growing interest and attention in Academia in Synthesis for advanced composite materials, particularly Structural and energy-based Systems/materials. The increasing number of publications in the years 2020 and 2025 reflects the attention these multifunctional materials with desirable mechanical properties, thermal stability, and environmental stability of the global research community. These materials are increasingly identified as having a vital role to play in innovation in clean energy systems, lightweight infrastructure, and high-performance engineering components. The present study contributes meaningfully to this evolving field by integrating experimental characterization, statistical analysis, and finite element modeling to investigate the mechanical behavior of hybrid nanoparticle-reinforced polymer composites. As a result, it supports both academic research and industrial use by providing validated insights that address two significant challenges in material design and predictive modeling. The bibliometric analysis highlights the manuscript's significance and contribution to the future development of sustainable structural materials by placing it in a broader scientific context.

4. CONCLUSION

In the present work, the mechanical performances of MWNT nanoparticles-reinforced PVDF/UPR composite were studied. It can be observed that the mechanical properties of composites, including tensile strength, flexural strength, hardness, and fatigue resistance, are noticeably improved with the incorporation of MWNTs. Besides, the optimal MWNT loading is found to be 3%. The uniform dispersion of MWNT in the PVDF matrix allows for an efficient improvement of the stress transfer between the components and a reduction in crack propagation. Consequently, the mode of failure has shifted from a more brittle nature to a more ductile nature. The experimental results were verified by FEA, and the maximum discrepancy was within 10%, proving that the computational models were quite accurate.

The results indicated that the addition of MWNT significantly improved the mechanical properties of PVDF/UPR composites and should be highly suitable for demanding industrial applications, especially aero-space, automotive, and structural engineering. The work provided new understandings of how MWNTs enhance the mechanical performances of PVDF-based composites, especially the optimal loading condition of 3%, which had not been considered highly in previous works. Future work on the effects of functionalized MWNTs can be directed at the interfacial bonding and testing the long-time durability of the composites resulting from cyclic load conditions.

5. AUTHORS' NOTE

The authors declare that there is no conflict of interest regarding the publication of this article. Authors confirmed that the paper was free of plagiarism.

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