Research Article

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Enhancing Mechanical, Moisture Resistance, and Fire-retardant Properties of *Nelumbo nucifera* Fiber/Epoxy Nanocomposites with Zinc Oxide Nanoparticles

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ABSTRACT

In recent years, an innovative material known as natural fiber-based nanocomposite has been developed. Furthermore, leveraging advancements in environmental nanotechnology, the eco-friendly composites exhibit potential for sustainable applications in lightweight structural materials and fire-resistant sectors, emphasizing reduced environmental impact and enhanced material efficiency. This investigation examined the influence of zinc oxide nanoparticles on moisture absorption, mechanical testing, fire retardant, and dynamic mechanical analysis of *Nelumbo nucifera* fiber (NNF) and epoxy (EP). The hand layup technique was implemented to create composite materials that comprised zinc oxide, NNF, and EP. The experiment revealed that the composites' modulus of elasticity (2117 MPa), tensile strength (48 MPa), and elongation at break (1.96%) were enhanced by increasing 12wt% zinc oxide (ZnO) nanoparticles. Conversely, the flexural strength and modulus of EP/NNF were 42 MPa and 2204 MPa, respectively. The modulus and flexural strength of EP/NNF/ZnO were 2814 MPa and 60 MPa, respectively, suggesting a 30.48% increase in bending strength. The swelling thickness and water absorption were substantially reduced (2.01% and 8.9%, respectively) as a result of the increased nanoparticle loading. To improve the dynamic mechanical properties (loss and storage modulus of 4.97 and 14.88 MPa) and performance (combustion rate of 18 mm/min, char residual of 31%) of composites, n-ZnO was incorporated. The X-ray diffraction (XRD) investigations showed that the 6 wt.% ZnO nanoparticles in the NNF fiber and EP samples still need to be more dispersed (2θ= 2.45º, intercorrelation of 24.52%, and d-spacing of 40.24 nm). The chemical composition and distribution pattern greatly impact the toughness. The combustion process is impeded by the fabrication of char shields with a higher concentration of nano zinc oxide, necessitating elevated temperatures for degradation.

Keywords: *Nelumbo nucifera* fiber; Zinc oxide; Flammability; XRD; Nanocomposite; Mechanical properties.

1. INTRODUCTION

In recent years, the increasing emphasis on sustainability and environmental consciousness has driven the development of advanced materials that are both eco-friendly and high-performing. Natural fibers, derived from renewable resources, have emerged as a viable alternative to synthetic materials due to their low cost, biodegradability, and favorable mechanical properties. These fibers, when reinforced in polymer matrices, provide a sustainable solution for various industrial applications, addressing the growing need for lightweight, durable, and environmentally responsible materials.

Natural fibers like lotus, palm, ramie, sun hemp, and spadix were characterized by their mechanical, chemical, and thermal properties. Lotus fiber displayed promising crystallinity and cellulose content, while palm fiber showed strong yield properties. These fibers offer sustainable alternatives for synthetic fibers with different industrial applications (Gokarneshan *et al.* 2021). The extraction of novel cellulosic fiber from the stem of *Nelumbo nucifera* (lotus) and assesses its characteristics. The fiber demonstrates a crystallinity index of 52.53% (XRD) and thermal stability around 210°C (TGA). Alkali-treated *Nelumbo nucifera* composite demonstrates significant potential for domestic applications (Elango *et al.* 2022). This research created a biodegradable packaging material from lotus fibers reinforced with banana stems, pineapple leaves, and rice straw, employing tapioca starch as a binder. The material underwent substantial biodegradation within 20 days, rendering it a feasible, environmentally acceptable substitute for plastic bags (Warrier *et al.* 2024). Lotus fibers were extracted and turned into yarns, then dyed wsith natural and chemical dyes. The fibers exhibited higher moisture absorption, lower crystallinity, and greater dye absorption compared to cotton. These properties make lotus fibers suitable for sustainable textile applications (Almas *et al.* 2024). Coiled lotus fiber yarn muscles showed exceptional tensile strength (38%)

and work capacity (450 J/kg), outperforming natural muscle fibers. (Wang *et al.* 2021). Reduced graphene oxide-coated lotus fibers (RGOLF) were engineered to exhibit strong electrical conductivity (4.63 μS) and superior sensing capabilities for NO_2 gas (Cheong *et al.* 2022).

Cellulase enzyme treatment improved the surface properties of lotus fibers, enhancing wettability, and antimicrobial properties. (Vajpayee *et al.* 2022). Lotus fiber composites with uniaxial, biaxial, and crisscross orientations show that uniaxial orientation provided the highest tensile and flexural strength. Alkaline-treated fibers and carbon fiber layers significantly enhanced the mechanical properties. (Thamba *et al.* 2023). Lotus fiber is highlighted for its mechanical strength, flexibility, and eco-friendly nature, with applications in textiles, healthcare, and aerospace. Challenges in production scalability and integration are discussed alongside the fiber's biodegradability and sustainable benefits, suggesting its future potential in various industries (Dhama *et al.* 2024). Sodium hydroxide treatment of micro-sized lotus fibers (MLFs) improved fracture toughness, increasing the critical stress intensity factor by 91% and Izod impact strength by 121%. Thermal and structural analysis validated enhanced biocomposite properties, optimized at 80°C, 6% NaOH for 6 hours (Trung, 2024). Lotus fibre/PVA/TiO_2 nanocomposites demonstrated superior thermal stability and photocatalytic efficiency, achieving 91.18% photodegradation of crystal violet dye. (Naveenkumar *et al.* 2023).

Lotus-blended fabrics exhibited improved air permeability, water vapor permeability, and thermal conductivity with reduced thermal resistance, making them ideal for summer wear. These properties highlight lotus fiber's potential in thermal management textiles (Ananthi *et al.* 2023). Cellulose/ZnO nanocomposites from lotus petioles showed outstanding antibacterial activity, achieving 100% inhibition of *S. aureus* and *E. coli*. The ZnO-decorated cellulose fibers offer promising applications in antimicrobial and environmental materials (Nguyen *et al.* 2024). The Lotus nanofibers, integrated into alginate porous membranes, exhibited improved mechanical properties (0.36 MPa, 17.7%), porosity, and in vitro degradation (59.2%), showcasing the potential for tissue engineering applications (Zhang *et al.* 2020a). Lotus fiber demonstrated superior absorption and desorption properties compared to cotton and flax fibers, with higher moisture regain and faster rates of equilibrium (Liu *et al*. 2020). The study investigated PVA nanofibers reinforced with nano-sized Ag, TiO_2, and ZnO, using SEM and TEM for morphology, FTIR for chemical interactions, and tensile testing for mechanical evaluation (Duygulu *et al.* 2024). Natural fiber composites with inorganic nanoparticles (e.g., TiO_2, MgO) were tested for tensile, flexural, impact strength, thermal stability, and water absorption. TiO_2 improved tensile strength by 90.43%, MgO enhanced flexural strength by 162.59%, and water absorption reduced by 36%. These enhancements make the composites suitable for lightweight and durable applications in various industries (Mishra *et al.* 2022).

Banana fiber composites treated with ZnO nanorods were analyzed using TGA, SEM, and mechanical tests. ZnO nanorods increased flexural strength (79 MPa for H2O2-treated fibers), reduced water absorption to 14.5%, and enhanced thermal stability. NaOH and $KMnO_4$ treatments yielded the highest improvements, supporting these composites for robust structural applications (Arumugam *et al.* 2021). A bilayer PLA-ZnO nanofiber membrane was fabricated for wound dressing applications and tested for mechanical properties, thermal stability, and water absorption (Hou *et al.* 2023). Sisal fiber composites with ZnO nanoparticles were tested for mechanical, acoustic, and thermal properties. Flexural strength peaked at 120 MPa, thermal conductivity improved with ZnO loading, and sound absorption efficiency was significantly improved (Swain *et al.* 2024). The research integrated 5 wt% of flame-retardant modifiers (EAD) into aramid fiber/epoxy composites, resulting in a limiting oxygen index (LOI) of 37.5% and self-extinguishing properties. Mechanical testing indicated a 17% enhancement in tensile strength and a 10% augmentation in ballistic performance (V50), broadening the composite's use in protective domains (Lan *et al.* 2024).

Kenaf fibers were coated with ZnO-TiO_2 nanoparticles to enhance flame retardancy and acoustic properties. The time to ignition doubled, and CO_2 emissions reduced significantly. Acoustic testing showed higher sound absorption, validating the coated fibers as eco-friendly, multifunctional composites (Samaei *et al.* 2022). Thermoplastic polyurethane reinforced with ZrP and ZnO nanoparticles showed improved flame retardancy, mechanical performance, and antibacterial activity. The PU3 matrix combined with 7% ZrP delivered superior tensile and impact strength, while 5% ZnO provided the highest antibacterial efficacy (Yaseen *et al.* 2024). Silk fabrics were coated with TiO_2, Al_2O_3, and ZnO nanofilms by atomic layer deposition. The nanofilms improved flame retardancy, enabling the fibre to self-extinguish, while maintaining intrinsic comfort and laundering durability, thus extending its service life (Zhang *et al.* 2024b). Kenaf fibers treated with ZnO nanoparticles improved hydrophobicity and mechanical properties in unsaturated polyester composites. The optimum ZnO content (2 wt%) enhanced water resistance, surface roughness, and interfacial bonding, validated by contact angle, FTIR, and SEM analyses (Mohammed *et al.* 2022).

The main objective of this study is to develop a hybrid composite material using NNF and epoxy (EP) with different amounts of nanozinc oxide by weight.

Researchers examined the effects of loading n-ZnO fillers on the mechanical properties of NNF-based hybridized epoxy composites. The objective of the investigation is to obtain the impact on the dynamic and overall mechanical properties of ZnO nanocomposites. Property that renders a substance combustible: Composite materials must possess fireproof characteristics. This study examines the impact of nano-ZnO infill loading on the inflammability properties of composites composed of hybridized epoxy and NNF. In order to evaluate the fire-resistant properties of the composites, heat release rate, and ignition duration are examined. This work has the potential to improve the field's knowledge of the impact of moisture absorption on the performance attributes of hybridized natural fiber materials and to facilitate the development of applications for moisture-laden and fire-resistant sectors.

2 MATERIALS AND METHODS

2.1 Materials

The nanomaterial utilized in this study was nano ZnO, characterized by a boiling temperature of 2360°C, a melting point of 1975°C, and a molecular weight of 81.38 g/mol. Moreover, they possess a density of 5.61 g/cm³. The epoxy (EP) polymer matrix has a density of 1.4 g/cm³, obtained from Kovai Cheenu Enterprises, Coimbatore, Tamilnadu, India was utilized as a binding agent. NNF is obtained from the local region of Erode, Tamil Nadu, India, and possesses an average length of 30 cm.

2.2 Fabrication of NNF Fiber

Fibers of *Nelumbo nucifera* are removed from the plant's stems by a meticulous process. The stems are severed, longitudinally bisected at 30 cm, and the internal fibrous threads are meticulously extracted. The fibers are subsequently cleaned to eliminate impurities and subjected to bleaching and alkaline treatment to improve their characteristics. The NNFs were purified and filtered prior to dissolution in distillation water at 73 °C, incorporating sodium chlorite (NaCl) (30 g) and acetic acid (10 mL). The system operated effectively for 60 minutes. The procedure was executed twice, incorporating glacial acetic acid and NaCl after each 60-minute interval. The system was agitated for 180 minutes following the final addition of the constituents. An alkaline solution was used to treat the fibers after rinsing and filtration in order to remove any residual acid. The fibers were cleaned to a pH of 7 by soaking them in a 5% NaOH solution, stirring them for 120 minutes at 70 °C, and then treating them in a vacuum chamber for at least one day at 70 °C. The processed fibers are now prepared for composite manufacturing. Post-treatment, the chemical composition of lotus fibers generally comprises 70–80% cellulose, markedly increased due to the elimination of non-cellulosic constituents. The hemicellulose concentration decreases to around 5–10%, but lignin levels reduce to about 2–5%. Minor constituents such as pectin and waxes are then reduced to 1–3%.

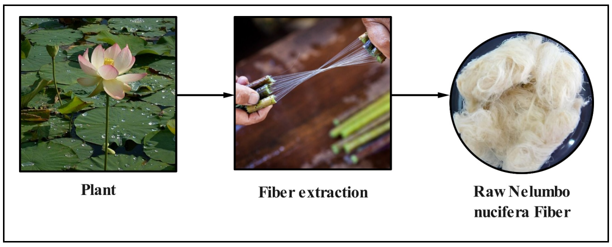

Fig. 1: Extraction procedure of *Nelumbo nucifera* fiber

2.3 Fabrication of Composite

The formulation of the EP/NNF hybrid blend is specified in Table 1. Nanomaterial integration was found to be at concentrations of 0%, 4%, 8%, 12%, and 16% based on the mass of dried NNF. ZnO nanoparticles are commonly utilized as nanofillers owing to their extensive surface area, a significant proportion, and compatibility with polymeric matrices. They provide composites exhibiting enhanced dimensional stability and heat resistance and mechanical properties. Nano ZnO can have functional groups or binding agents added to its surface to make it more dispersed and compatible with polymeric matrices. ZnO nanoparticles were dispersed in epoxy matrix and then it was stirred using mechanical stirrer for 15–30 minutes. This enhances ZnO-polymer interaction, leading to improved mechanical properties and fire resistance. All formulations were designed with a 4% maleic anhydride grafting binding reagent concentration, in line with the polymeric substrate. It was possible to create five different sets of hybrid and combined specimens.

Composite specimens were fabricated by adding NNF into an epoxy resin matrix. The experiment was conducted with the hand layup technique. The hand layup method, widely used for composite fabrication due to its simplicity and cost-effectiveness, significantly influences the overall properties of the ZnO-reinforced NNF/EP composite. This method allows for easy incorporation of natural fibers and nanoparticles, enabling the development of customized composites with controlled mechanical and fire-resistant properties. The hand layup method impacts the composite's properties due to its manual nature, affecting fiber distribution, resin impregnation, and void content.

A different percentage of ZnO nanoparticle compositions were employed in the production of NNF fiber-reinforced epoxy hybrid composite specimens. The fabrication processes are as follows. Initially, the mold was developed using wax, which functioned as a releasing agent. Subsequently, in accordance with the provided directions, the epoxy was combined in a 10:1

ratio. The hardener was advantageous as a curing agent for anhydrous bases. The ZnO was included in the epoxy resin as a flame retardant after the substance was meticulously mixed. A mechanical stirrer agitated the epoxy resin and silica slurry at 1200 rpm for 5 minutes to prevent accretion resulting from the chemical reaction. Epoxy resin was then applied to the surface of the mold. The mold was filled with epoxy and NNF fiber in a haphazard configuration. The mixture was subsequently poured into the mold to create the specimens' batter surfaces. A supplementary steel plate was employed to compress the composite, which was first encased in a plastic covering to prevent adhesion to the mold plate. The specimen was allowed to mold at room temperature for 24 hours. A specimen was obtained from the mold following its desiccation. Thereafter, the mold was sanitized in anticipation of the forthcoming testing in accordance with ASTM standards. The weight percentage of the NNF fiber is consistently maintained at 35wt%, while the required amounts of epoxy and ZnO to fill the mold were established by calculating the masses of the fiber and its binder for each sample. Table 1 presents the material composition of the sample, while Fig. 2 illustrates the fabrication process of the composite samples.

Table 1. The material composition utilized in this study

Symbols Code	Epoxy (wt.%)	NNF (wt.%)	Nano ZnO Particles (wt.%)	Theoretical density	Actual density	Void
S1	65	35	0	1.694	1.612	4.841
S2	61	35	4	1.720	1.637	4.815
S3	57	35	8	1.748	1.665	4.748
S4	53	35	12	1.776	1.692	4.726
S5	49	35	16	1.805	1.720	4.698

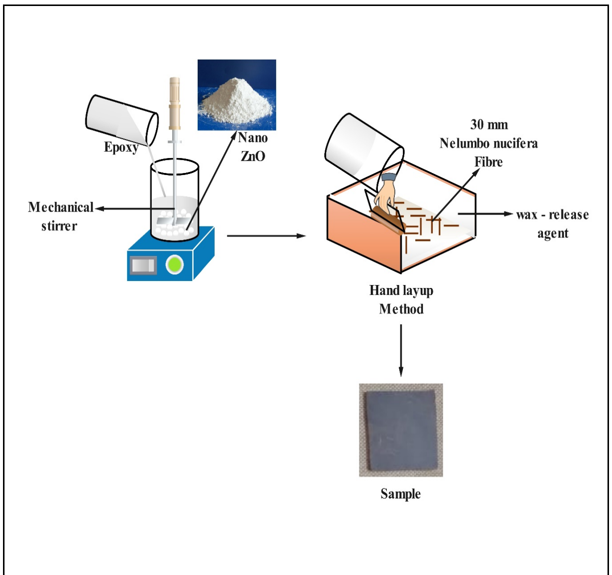

Fig. 2: Experimental process of this study (Hand layup)

2.4 Analysis of Composites

2.4.1 XRD Analysis

The XRD technique was employed to assess the dispersion of nanoparticles in EP/NNF hybrids. Assessments of ZnO Nanopowder and its constituents were performed by X-ray diffraction (XRD) utilizing a CuKα radiation (2θ is 1.50 nm). The power supply supplied was set to 50 kV and 50 mA.

$$\text{Crystallinity Index (CI)} = \frac{I_{200}-I_{\text{in}}}{I_{200}} \quad \dots (1)$$

Equation (1) shows the crystallinity index that denotes the relative degree of crystallization, I200 indicates the intensity of the peak (200) planes, and Iin reflects the intensity of the scattered peak at 2ϴ = 18°, which was employed to calculate the CI. The scanning phase proceeded without interruption, happening at a pace of 1º per minute, within a range of 0 to 15º. The broken surface of the sample was analyzed utilizing a scanning electron microscopic (SEM) analysis (Carl Zeiss, Supra 55).

2.4.2 Mechanical Testing

Tensile tests evaluating strength, elasticity, and elongation at break were performed utilizing an Instron universal testing machine (UTM) at 2 mm/min crosshead speed. Flexural tests were performed utilizing a 3-point bending test with respect to the ASTM D 790 standard, employing a 2 mm/min crosshead speed and a lengthy span of 50 mm. Each combination was assessed through three instances, and the average findings were reported.

2.4.3 Fire Properties and Dynamic Mechanical Analysis (DMA)

Experiments were performed through a cone calorimeter with a quick heat transfer of 50 kW m^{-2} to assess the combustion characteristics in accordance with ASTM E1354, encompassing char residues, complete smoke production, and duration of combustion. The combustion rate of the materials was evaluated in accordance with ASTM D635. A computerized 615 DMA analyzer has been employed to assess the substrate's DMA at different temperatures. A strain intensity of 0.2 mm and 1 Hz frequency were used to study the dynamic properties during the constant frequency phase. Within the temperature range of -30ºC to +180ºC, the specimens were steadily heated at a rate of 10 ºC/min. At each treatment level, three separate specimens were tested for each testing.

2.4.4 Moisture Absorption

The moisture absorption and thickness swelling tests were conducted in accordance with ASTM D7031. The evaluation of water absorption was conducted in two stages. The specimens were produced, dehydrated in the oven at 100 ºC for 24 hours until a stable weight was achieved, and subsequently saturated with fluids. The samples were fully submerged in water at ambient temperature following saturation. The specimens were measured and weighed after the exterior liquid layer was removed after 30 days of collection to ascertain their measurements.

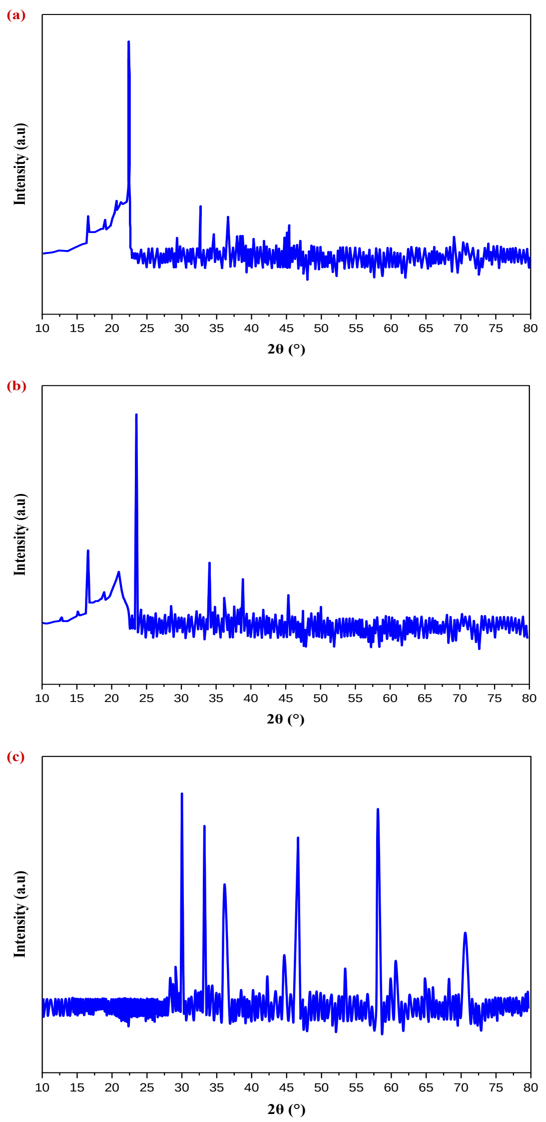

Fig. 3: (a) Raw fiber; (b) Treated fiber; and (c) addition of nano ZnO with treated NNF hybrid composites of XRD analysis

3 RESULT AND DISCUSSIONS

3.1 XRD Analysis

Chemically modified and untreated NNF fibers' X-ray scattering patterns are shown in Figure 3. Cellulose molecules on surfaces produce peaks at approximately 2θ = 19.41, 24.98, and 37.65º, respectively. The existence of type I cellulose in the fibers is illustrated in Figures 3 (a) and (b) before and after the bleaching operations. The maximum intensity of the 200 reflections has risen, signifying an improvement in the level of crystallization. The alkaline treatment expedited the transformation of cellulose Iß to cellulose Iα, as evidenced by the modest change in 2ϴ from 25.41 to 25.48°. This occurred even with minimal NaOH concentrations, such as those used in this investigation. The raw and chemically processed fibers have crystallization indexes of 56% and 68%, respectively. According to the results, the bleaching procedure improves the crystalline structure of NNF fibers, which may clarify their use in polymeric mixtures. Fig 3 (c) illustrates an outcome of the XRD analysis of the ZnO. A sample of ZnO spherical particles exhibits a diffraction circle analogous to a glassy phase at low angles alongside a ZnO crystalline phase, with the most pronounced diffraction peak occurring at approximately 31.74°. The crystalline structure of ZnO promotes its durability and stability, which are essential for its optimal performance. XRD and SEM characterized zinc oxide nanoparticles synthesized using Timla leaf extract. The nanostructures demonstrated potential for integrating with natural fibers, providing green and sustainable solutions for functional material development (Kour *et al*. 2021)

XRD was utilized to analyze the skeletal state of the composition. The interlayer spacing in ZnO Nanoparticles (according to Bragg's law) and the inter-correlation coefficient (I) of the polymers in nZnO were quantified using specific methods to verify a uniform distribution of nanomaterials inside a polymer matrix.

$$n\,\partial = 2d\mathrm{Sin}\ \theta \qquad \ldots (2)$$

$$\text{Intercorrelation } = \frac{d-d_0}{d_0} x100 \qquad \ldots (3)$$

In this context, d_0 refers to the spacing of the clean ZnO filled, n denotes the integer, ∂ signifies the diffraction wavelength, d indicates the internal coatings of ZnO, and ϴ denotes the angle of diffraction.

The d-spacing and I values of the nano ZnO in the polymeric composite are presented in Table 2, as determined by formulae (2) and (3). The sequence of integration was shown to increase and then decrease as the particle concentration was increased to 12% in this table. The specimen with 12% nano ZnO's peak was shifted to a lower orientation with a 12% concentration of nanoparticles, signifying the development of an interwoven structure and enhanced dispersion. This is a result of the limited application of the bonding agent in the nanocomposites. The basal separation of ZnO particles can be improved, and the penetration of polymeric chains into them can be more efficient by augmenting the affinity of the polymer matrix with ZnO. Alkaline ammonium surfactants modified nanofiller materials effectively promote the synthesis of nanocomposite materials in polymers containing polar functional groups. Subsequent experiments revealed that a rise in particle addition resulted in a larger aggregate size of the dispersed n-ZnO, which occasionally coalesced. In contrast, the presence of a binding agent improved the distribution of ZnO by facilitating the disintegration of agglomerates into smaller clusters.

Table 2 The intercorrelation and d-spacing values of nano ZnO/NNF/PP hybrid composites at varying weight percentages

S. No.	Specification	2θ (°)	d-Spacing (nm)	Intercorrelation (%)
1	4wt% of ZnO	2.61	33.62	7.56
2	8wt% of ZnO	2.54	35.78	15.27
3	12wt% of ZnO	2.32	39.01	23.41
4	16wt% of ZnO	2.37	38.52	20.69

3.2 Mechanical testing

3.2.1 Tensile properties

The stress vs strain curve of the tensile characteristics is depicted in Figure 4. Among the various weight proportions of ZnO particles, the 12 wt.% nZnO composites exhibit the highest Young's modulus and endure the greatest tensile stress. The reduced diffusion levels that result from the considerable aspect ratio of nZnO are the cause of the enhancement of properties at low-weight nanolayered zinc oxide loading. The enhancement in properties may also be attributed to the formation of integrated and refined polymeric nanostructures at these zinc oxide concentrations. The formation of aggregated ZnO fillers may be linked to the reduced tensile properties that occur with increased weight application.

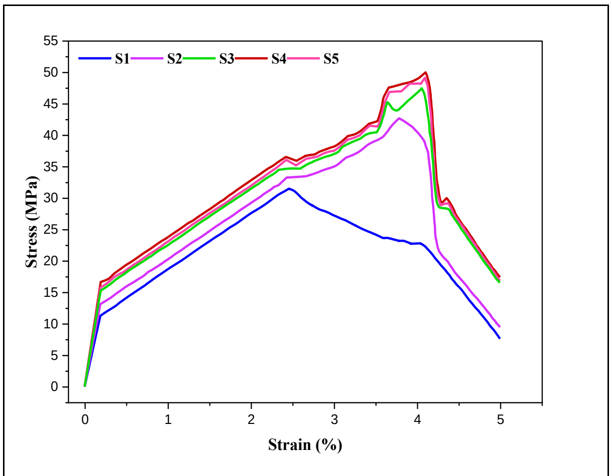

Fig. 4: Under tensile loading, the stress-strain curve of nano ZnO/NNF/EP-based hybrid composites with varying weight percentages is presented

Additionally, the nanoparticles' ability to enhance one another is reduced by two distinct mechanisms. The dispersion of nanoparticles across the polymer-based laminate surfaces is the cause of their adverse effect, which results in reduced performance. The reinforcement of ZnO may be reduced by the accumulation of nanomaterials at a concentration of 20 wt% of ZnO.

The effect of ZnO nanoparticles on the strength characteristics of EP/NNF mixes was found to be substantial, according to the present investigation. The researchers' findings indicate that adding an extra reinforcing filler improved the young modulus of the composite materials. The addition of NNF is well acknowledged to augment the stiffness of the hybrid while diminishing its flexibility. This is particularly applicable to polymer composites, as the incorporation of fillers restricts the mobility of polymer chains, hence enhancing the material's elasticity. The tensile properties increase with particle concentration up to 12%, beyond which they decline, as illustrated in Fig. 5. TiO_2 composites exhibited maximum tensile strength (90.43%), Al2O3 provided the strongest impact resistance (17.3 J/cm²), and water absorption reduced by 36% reinforced with Jute and banana fiber composites (Tasnim *et al*. 2024).

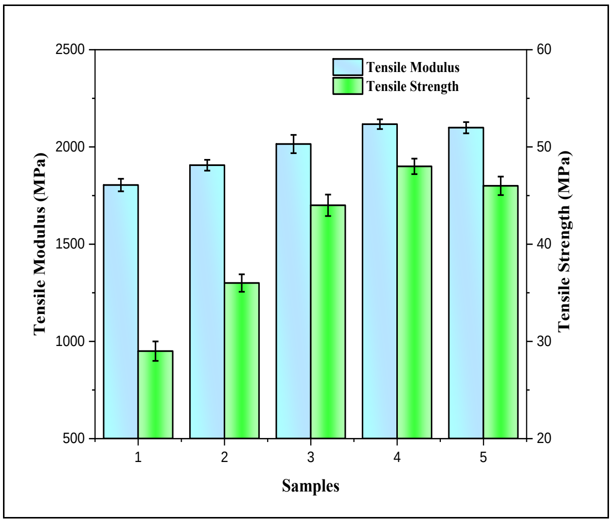

Fig. 5: The modulus values and tensile strength of hybrid composites based on nano ZnO/NNF/EP at varying weight percentages

3.2.2 Flexural Strength

Figure 6 illustrates the stress-strain curve under flexural loading. Analysis of the flexural characteristics of n-ZnO and EP/NNF composites is shown in Figure 7. The 12 wt.% ZnO filler exhibits the highest flexural modulus and sustains the greatest bending force among various ZnO filler concentrations. The modulus and bending strength of pure EP/NNF were 42 MPa and 2204 MPa, respectively. In contrast, the flexural strength and elasticity of EP/NNF/ZnO were 60 MPa and 2814 MPa, respectively, indicating a 23.5% rise in flexural stiffness.

Figure 7 demonstrates that the flexural strength and modulus enlarged with the addition of additional ZnO (12 wt%) in comparison to the EP/NNF. The flexural capacity primarily depends on the fiber, and NNF exhibits superior bending properties compared to clean EP.

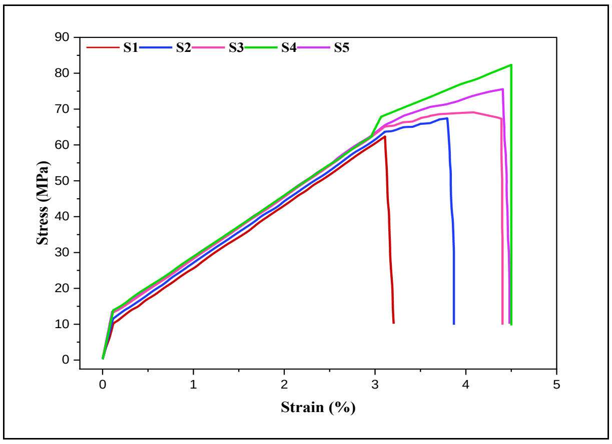

Fig. 6: Flexural loading stress versus strain curvature of different wt.% of nano ZnO/NNF/EP based hybrid composites

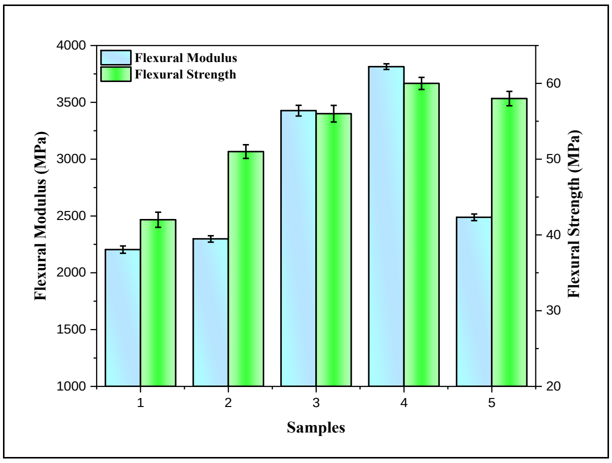

Fig. 7: Flexural properties of different wt.% of nano ZnO/NNF/EP-based hybrid composites

The interfacial bonding between the epoxy and the fibers influences the documented improvements in mechanical efficiency, resulting in improved load transfer and improved flexural performance. At the maximum concentrations of ZnO (10 wt% and 8 wt%), the reduction in strength and elasticity was 67% and 35%, respectively. The decline may be attributed to the inferior ZnO generated during its fabrication as an outcome of the extraction of naturally occurring components. The secondary source of these reductions is the issue of ZnO scattering in the EP substrate. The flexural properties are influenced by the accumulation that results from insufficient dispersion, as they are contingent upon the matrix. Premature failure and ineffective load transmission may result from substantial aggregation at the interface of EP, NNF, and ZnO. ZnO nanorods enhanced stiffness by 58% flexural and 31% tensile but reduced thermal stability due to PLA degradation (Sbardella *et al.* 2021).

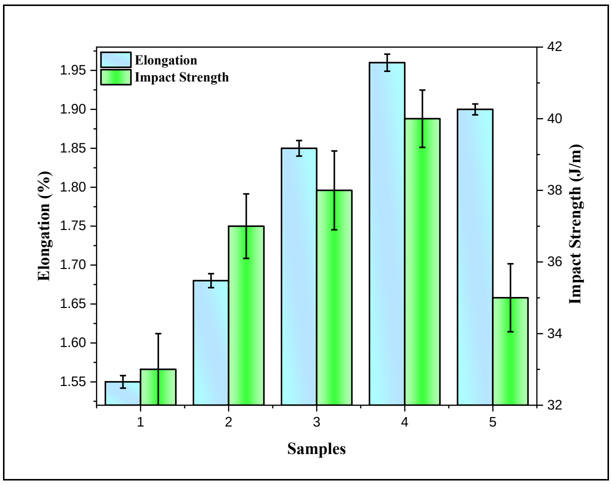

Fig. 8 Evaluation of Impact strength and elongation values of various hybrid composites

Table 3. Results on mechanical properties of hybrid composites

Samples	TS (MPa)	Young' s Modules (MPa)	FS (MPa)	Flexural Modulus (MPa)	% of Elongation	IS (J/m)
1	29	1804	42	2204	1.55	33
2	36	1906	51	2298	1.68	37
3	44	2015'	56	2427	1.85	38
4	48	2117	60	2814	1.96	40
5	46	2099	58	2488	1.9	35

3.2.3 Impact Strength

The impact resistance of the laminate would decrease as the content of NNF fibers increased. Minor fractures developed at the impact location because of the insufficient interaction between NNF fiber and EP, facilitating the propagation of fractures throughout the mixture. Fig 8 illustrates that when the concentration of nanoparticles escalates, the impact on the strength of the nanocomposite wanes. At higher concentrations of nanolayered zinc oxide, the impact resistance decreases due to ZnO agglomerates and unexfoliated aggregates as voids. Subsequent testing revealed that an increase in the quantity of reinforcing filler resulted in a decrease in the elongation at the break of hybrids. The elasticity of the EP/NNF fiber hybrids was markedly reduced with increased filler loading. In addition, the elongation at break increases as the nanomaterial content rises to 12wt%, after which it starts to decrease. Mechanical properties improve up to 12 wt.% ZnO due to effective stress transfer, enhanced fiber-matrix bonding, and uniform dispersion. Beyond this, agglomeration and phase separation occur, creating stress concentration points, poor bonding, and void formation, leading to weaker mechanical performance. The elevated concentrations of nano ZnO account for this changes in elongation. Table 3 presents the specifics regarding the comprehensive mechanical properties of nano ZnO/NNF/EP-based hybrid composites.

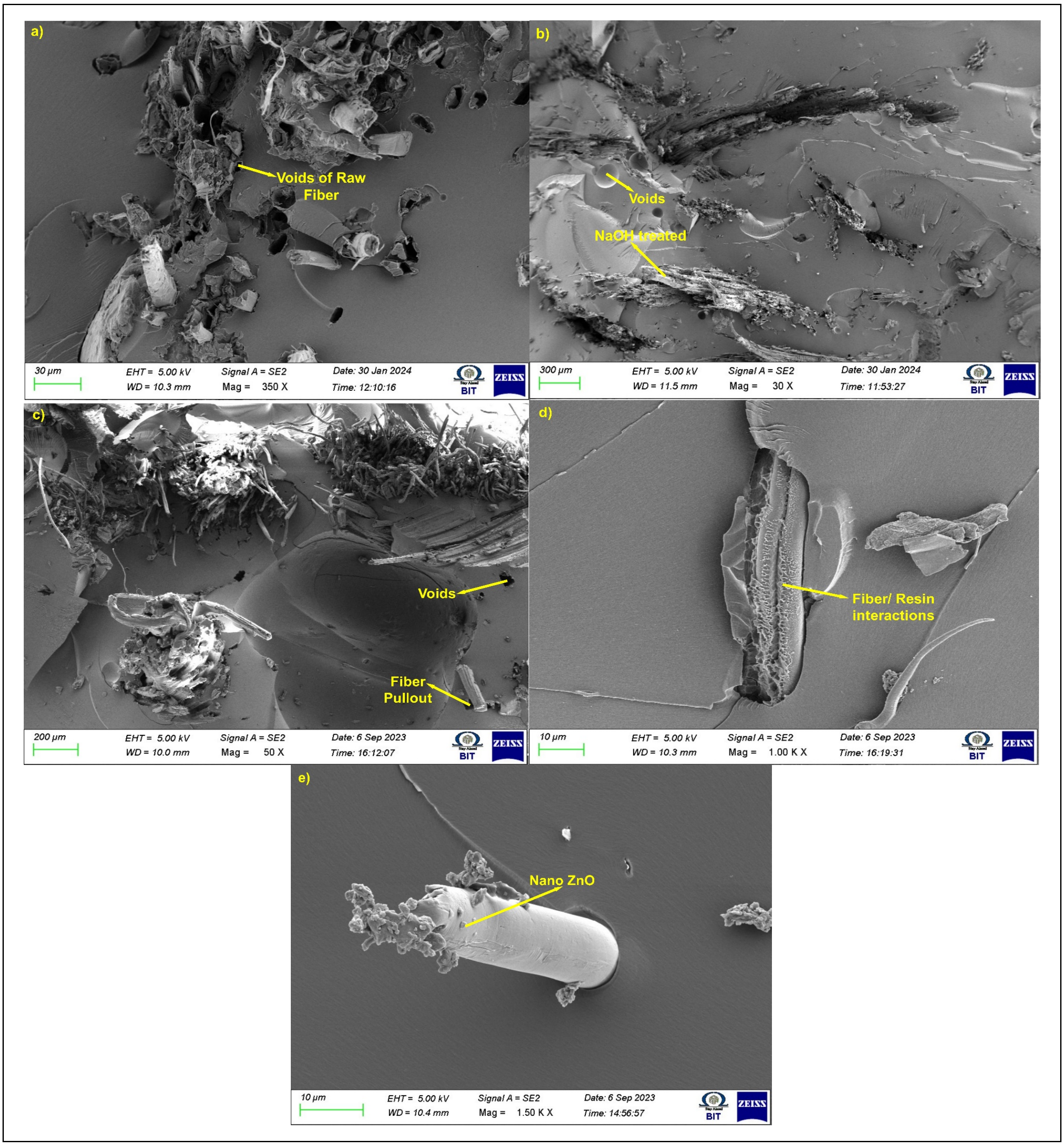

Fig. 9 SEM images of sample (a) S1 at 0w% ZnO, (b) S2 at 4%ZnO, (c) S3 at 8%ZnO, (d) S4 at 12%ZnO and (e) S5 at 16%ZnO

3.2.4 Microstructural Analysis

Subsequent to mechanical testing, the cracked surfaces of the EP/NNF and hybrids containing ZnO particles were analyzed, as depicted in Fig. 9. Fig 9(a) denotes that the NNF-reinforced EP displays fiber pullout or other indications of inadequate interfacial bonding. Figure 9(b)–(e) depicts the tensile fractured behaviors of hybrid composites using nano-ZnO fillers at varying weight percentages. The EP and NNF matrices displayed a consistently disrupted interface, whereas the matrix of the minute materials revealed a rough fracture look. The addition of 12 wt% ZnO to the base material effectively achieved a homogeneous dispersion of nanoparticles within the EP-based substrate, as illustrated in Figure 9(d). Nevertheless, the large-scale fiber pullout and deformation that occurred suggest a feeble connection between the matrix and the fibers. As illustrated in Fig. 9(c) and (e), the hybrid laminates exhibited conspicuous aggregation as the infill content increased in the base material. SEM study indicated aggregation and irregular distribution of inorganic oxides, which boosted functioning (Bajwa *et al.* 2024).

The fracture morphology of EP/NNF/nano ZnO-based hybrid composites following flexural testing is illustrated in Figures 9(d) to (e). The adhesion of natural fibers to the matrix in the composite is crucial for optimal mechanical stability and load transfer. The fibers can be readily detached from the substrate as a result of poor adhesion, which can result in fiber pullout during flexural stress (Fig. 9(e)). ZnO nanoparticles are thought to enhance the bonding between the epoxy and fibers in composite materials. The SEM image of the material (Fig. 9(d)) indicates a homogeneous and uniform distribution of 12 wt.% ZnO nanoparticles, demonstrating that the particles were effectively integrated and dispersed inside the epoxy matrix. This effective distribution enhances the interfacial bonding among the NNF fibers and the polymeric composites, leading to superior binding. Regions with elevated fiber concentrations or clustering may result from improper fiber orientation or aggregation.

As a consequence of these congested regions, void formation may occur and proper matrix flow may be impeded. In regions where the fibers are densely packed or are distributed unevenly, the SEM image may indicate the possibility of cavity formation. If the substrate appears to completely encapsulate the fibers, with no discernible gaps or distortion, an elevated fiber-matrix attraction was recommended. The robust adhesion arises from the effective hydration of the matrix, the suitable surface treatment of the fibers, and the 12wt% of ZnO nanoparticles that enhance interfacial interactions. In the aggregated regions, the polymers may undergo rapid degradation as a consequence of the highest stress accumulation. An EP matrix with uniformly distributed ZnO filler is purported to provide superior mechanical characteristics. Cotton fibers were functionalized with ZnO and copper nanoparticles using ultrasound waves and citric acid as a binder. The treatment altered fiber morphology and enhanced cellulose crystalline phases (Silva *et al.* 2022).

3.3 Dynamic Mechanical Analysis (DMA)

The DMA performance of composites composed of EP and NNF fiber is illustrated in Figure 10, which illustrates the influence of nanoparticles (ZnO). The DMA loss and storage modulus values increased, despite the increase in the quantity of nano-ZnO. The configuration of the system influences the DMA properties of hybridized composites, the characteristics of the individual components, and the character of the interactions between the minute particles and polymeric matrix. The intermediate phase is initiated by the addition of minute particulates into a substrate to produce polymer composites. The stability of nZnO and the matrices is mirrored by the modification of the surface related to nZnO. It exerts a considerable influence throughout this interphase interval. By increasing the weight percentage of the nanofiller by 16%, the dynamic mechanical characteristics of the composites were improved.

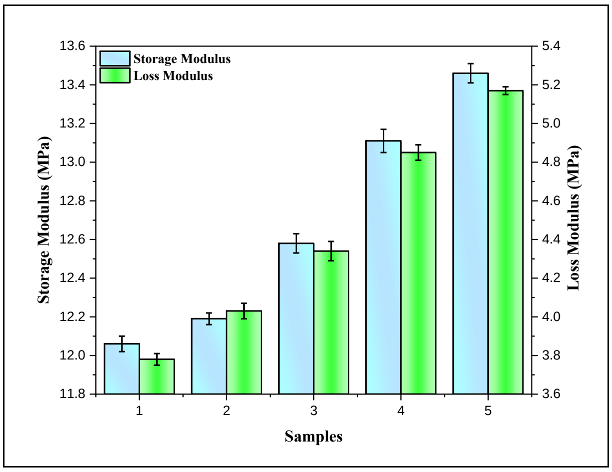

Fig. 10: DMA characterization of hybrid composites founded on nano ZnO/NNF/EP at varying weight percentages

3.4 Flame Retardant Properties

A diverse array of flammable compounds is utilized on different materials to inhibit combustion or retard fire progression. The emphasis is on the molecular mechanism of action rather than their physical characteristics. Flame retardant chemicals are under investigation due to their widespread use and concerns over potential adverse health consequences. Fig. 11 depicts the augmentation of char deposits in relation to the escalating concentration of micro ZnO. Further investigation indicated that a layer of carbonaceous zinc oxide char had formed on the surface of the nanocomposite before the fire event. The inorganic-rich outer layer exhibited superior barrier characteristics, hence substantially decreasing the combustibility of the hybrid. The synthetic layer greatly reduces the igniting time of these materials with a low concentration of multilayer zinc oxide nanoparticles because it acts as a great insulator for heat and oxygen transport. Moreover, Fig. 11 demonstrates that an elevation in micro ZnO concentration is associated with a decrease in total smoke output. The thermoregulation of the nanomaterials is impeded by the ceramic coating on the specimens' surfaces, which is widely believed to have developed as a result of the condensation of artificial nZnO dispersed-layered zinc oxide membranes within the cone calorimeter. Smoke reflection is reduced by a small quantity of nanoparticles, which enhances transparency from all angles and aids humans in evacuating flames.

Also, it's important for polymer composites because it reduces the amount of toxic smoke.

The ZnO nanoparticles enhance fire retardancy by forming a dense, compact char layer that acts as a thermal and oxygen barrier. SEM analysis of the burnt residues reveals that at higher ZnO concentrations (up to 16 wt.%), the char layer becomes more structured and interconnected, improving thermal stability and reducing combustion rate (18 mm/min). The char composition consists of carbonaceous residues and ZnO-ceramic components, which contribute to a ceramic-like protective shield, slowing down heat transfer and material degradation. Additionally, the char layer helps trap decomposed gases, reducing smoke emission and improving fire safety. With 31% char residue formation at 16 wt.% ZnO, the composite exhibits enhanced flame resistance by delaying ignition and acting as a strong insulating barrier.

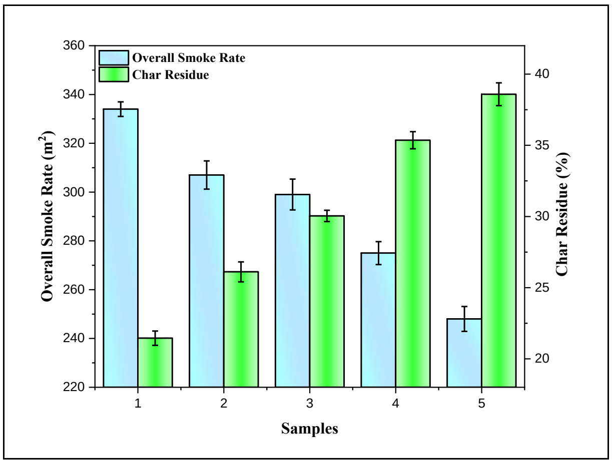

Fig. 11: The overall combustion rate and char residue of hybrid composites based on nano ZnO/NNF/EP at varying weight percentages

Combustion rates of the blends decrease with increasing proportions of nanomaterials, as seen in Fig. 12. Enhancing fire-retardant properties with minimal ZnO emissions necessitates the procurement of increased quantities of nZnO. Nanocomposites made with ZnO dispersion are intended to have better flame retardant capabilities. The effective carbonaceous-silicate char formed on the surface during combustion imparts fire-retardant characteristics to the nanocomposites, safeguarding the underlying material and reducing the mass loss rate of degradation products. Fig. 12 shows that the hybrids' ignition time is affected by the concentration of ZnO. Results for nanocomposites' flammability and thermal stability were also quite similar. The char barrier's longevity is greatly affected by the chemical formulation and distribution. Producing char shields with large concentrations of microzinc oxide makes combustion more difficult and degradation more temperature-intensive. Cotton fabrics were modified with formulations of chitosan, thyme oil, and flame-retardant fillers (SiO_2, ZnO, TiO_2, LDH). The Cotton fabrics exhibited excellent antimicrobial activity and reduced flammability, with the LDH and TiO_2 fillers yielding the highest reductions in heat release rate and microbial growth inhibition (Szadkowski *et al.* 2023).

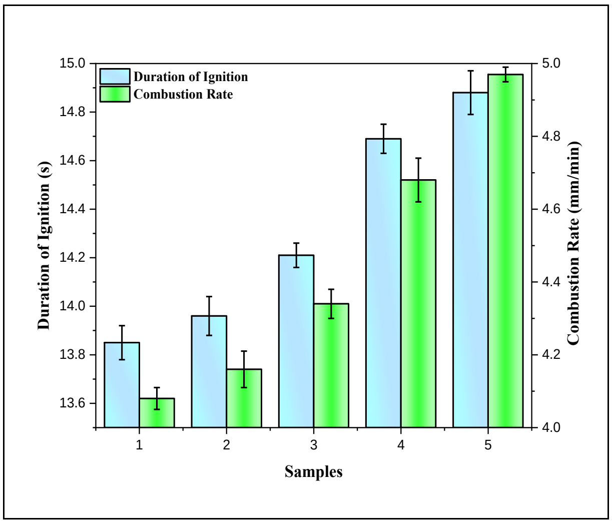

Fig. 12: Burning rates and duration of various weight percentages of nano ZnO/NNF/EP-based hybrid composites

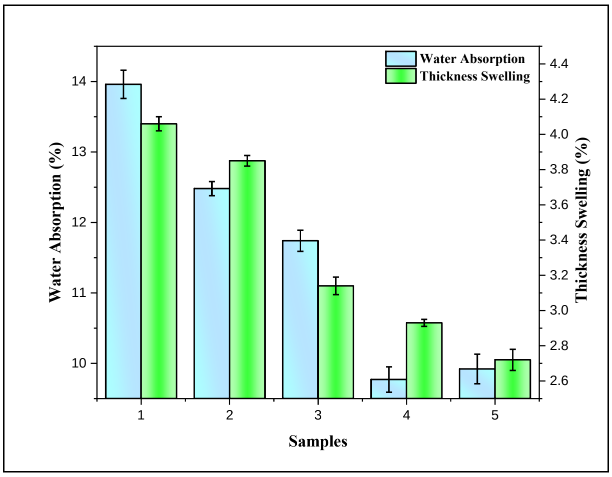

Fig. 13: The moisture absorption properties and thickness swelling of hybridized composites based on nano ZnO/NNF/EP at varying weight percentages

3.5 Moisture Absorption Properties

The limited water absorption capacity of cellulose fibers results in adverse impacts on the dimensional and structural integrity of natural fiber plastic composites when they are exposed to moisture. To comprehend the application-based materials' long-term durability, it is crucial to investigate the moisture absorption and retention characteristics of the organic fiber composites. Atomic water can enter fiber composites primarily through capillary action, diffusion through micro-gaps in the polymer chains, and defects in the filament-matrix link. Hydrogen bonding is the main mechanism by which the surface and underlying structure of the fiber absorb moisture.

Moisture penetration and thickness swelling are diminished as the concentration of nanolayered zinc oxide increases, as illustrated in Figure 13. It appears that the repelling properties of nanoparticles prevent water from permeating the polymer matrix. Two processes have been identified as responsible for this behavior. The primary factor is the hydrophilic nature of ZnO, which tends to retain moisture, coupled with the challenge posed by surfactant-coated ZnO nanoparticles for liquid mobility. The composite's barrier characteristics inhibit water ingress into its interior. It seems that one of the previously stated approaches may be more efficacious when the structure is treated. The reduced surface area of nano ZnO, characterized by its hydrophilic nature, along with the complex pathways of surfactants in the deleted structure, results in diminished fluid movement under severe conditions. ZnO nanoparticles were incorporated at varying concentrations (0–3%), improving physical properties such as density, thickness swelling (38% reduction), and water absorption (12% reduction) (Gul *et al.* 2021).

The reduced water absorption and swelling thicknesses may be explained by the alteration in the crystallization of polymer mixtures associated with the binding agent and the use of zinc oxide nanoparticles as nucleating agents. The crystal structure of binder-modified composite polymers is significantly preferable to that of comparable untreated polymeric mixtures. The crystalline structure of the composite material and the production efficiency of nuclei can be improved by the addition of fine particles as a nucleating agent. These materials demonstrate diminished liquid absorption as a result of the impermeability of the crystallization regions.

4. CONCLUSIONS

The following results were derived from the synthesis of organic NNF with different weight ratios of nano ZnO-based epoxy hybrid and the assessment of their structural, mechanical, flammability, DMA, and moisture absorption properties.

- The occurrence of type I cellulosic in the fibers is indicated by XRD, regardless of whether the bleaching operations were performed prior to or subsequent to the analysis. The degree of crystallization has been improved, as evidenced by the increase in the maximum intensity of the 200 reflections. Furthermore, the minor alteration in 2ϴ ranges from 25.41 to 25.48°. The apex of the specimen with 12% nano ZnO concentration shifted to a lower position, signifying the formation of an intertwined structure and enhanced dispersion.
- The mechanical characteristics are enhanced by an increase in ZnO particle concentration of up to 12%, after which they decline. The diminished diffusion rates that result from the considerable aspect ratio of nano ZnO are the reason for the enhancement of properties at minimal weights of nanolayered zinc oxide loading.
- The flammability characteristics indicate that an increase in nano ZnO content (16 wt.%) results in a reduction of overall smoke emission. It is generally believed that the ceramic coating that prevents the nanomaterials from being thermoregulated was generated by the condensation of synthetic nano-ZnO dispersed-layered zinc oxide membranes within the calorimeter's cone. This coating is applied to the specimens' surfaces.
- The repelling properties of the 12 wt. % nanoparticles impede the infiltration of water into the polymer matrix. This behavior has been ascribed to two processes. The primary factor is the hydrophilic nature of ZnO, which generally holds moisture, coupled with the observation that ZnO nanoparticles coated with surfactants obstruct liquid transport.

The use of ZnO nanoparticles and NNF fibers offers several environmental benefits, aligning with sustainability goals. NNF fibers are renewable, biodegradable, and derived from natural sources, reducing dependence on synthetic reinforcements. Their low energy processing and minimal carbon footprint make them an eco-friendly alternative to traditional fibers like glass or carbon.

To optimize the mechanical and fire-retardant properties of the ZnO-reinforced NNF/EP composite, ultrasonication and high-shear mixing can improve ZnO dispersion, while silane coupling agents can enhance matrix compatibility. Hybrid reinforcement with TiO_2, graphene, or jute fibers can boost tensile and flexural properties, and vacuum-assisted resin transfer molding (VARTM) or compression molding can reduce porosity and improve adhesion. For enhanced fire resistance, intumescent flame retardants or ceramic coatings can stabilize the char layer, while long-term durability studies and self-healing coatings can improve structural longevity.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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