



Development of Nanoparticle-enhanced Luffa Fiber Bio-epoxy Composites with Improved Thermo-mechanical Performance

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ABSTRACT

In the present investigation, bio-composites consisting of luffa fiber reinforced bio-based epoxy matrix (LE) incorporated with various nanoparticles was fabricated using a hybrid processing technique that integrates vacuum-assisted resin infusion with autoclave curing. Carbon-based, metal oxide, and layered silicate nanoparticles were utilized to evaluate their influence on mechanical behavior of the bio-luffa composites. The nanoparticles were synthesized and subsequently subjected to heat treatment based on their material characteristics. Fourier Transform Infrared (FTIR) analysis verified the presence of corresponding functional groups. Dynamic mechanical analysis showed that storage modulus at glassy area increased from about 2200 MPa for the neat LE composite to approximately 3750MPa, 3050MPa, and 2650MPa for LE–ZrO₂, LE–MMT clay, and LE–TiO₂ nanocomposites, respectively. In the rubbery plateau region at around 100°C, the modulus increased from ~230MPa to about 540MPa, 590MPa, and 450MPa for the respective nanocomposites, indicating enhanced stiffness and improved filler–matrix interactions. Furthermore, glass transition temperature shifted from ~70°C for neat composite to about 82°C and 84°C for LE–ZrO₂ and LE–MMT nanocomposites, respectively, demonstrating restricted polymer chain mobility due to stronger interfacial bonding. Nanofillers possessing lamellar structures, such as layered silicates, exhibited a more pronounced effect on toughness of the bio-luffa composites compared with the particle-shaped morphology of metal oxide nanoparticles. Although micro-voids of varying sizes were observed through scanning tomography, these defects did not significantly affect the mechanical performance of composites, indicating that reinforcing action of nanoparticles remains governing mechanism determining overall mechanical response of bio-luffa composite system.

Keywords: Dynamic mechanical analysis; Storage modulus; Montmorillonite; Heat treatment; Nanocomposites; Luffa fiber.

1. INTRODUCTION

The advancement of eco-friendly composite materials has emerged as a focal point in Engineering and materials research in recent years. Epoxidised oil-based resins exhibit good compatibility with natural fibers and nanofillers, providing improved mechanical strength, thermal stability, and reduced water absorption, demonstrating their potential as a sustainable alternative to petroleum-based polymers (Baishya *et al.* 2026). Vegetable oil extracted from kapok seeds was epoxidised by performic acid to produce epoxidised kapok seed oil (EKSO) with an oxirane oxygen content of 2.7%. The epoxidised oil demonstrated good performance as a co-stabilizer for PVC, enhancing its thermal stability while

maintaining acceptable mechanical properties (Putrawan *et al.* 2023). Epoxidised vegetable oils derived from rapeseed oil and castor oil were synthesized and further modified through ring-opening reactions with polyethylene glycol. The resulting polymeric vegetable oils showed good emulsifying properties and were successfully applied as environmentally friendly emulsifiers for metal-working cutting fluids (Abd El-Rahman *et al.* 2021). Hybrid bio-composites were fabricated using alkali-treated luffa and jute fibers in different blend ratios through compression molding with epoxy resin. The composites exhibited improved mechanical and dynamic mechanical properties with higher jute content, while increased luffa content enhanced acoustic performance, demonstrating their

potential for load-bearing and sound absorption applications (Suba and Boominathan, 2024).

Kevlar/luffa fiber sandwich composites reinforced with graphene nanoparticles were produced to evaluate their mechanical, viscoelastic, and impact resistance properties. The incorporation of 3 wt% graphene significantly enhanced tensile, flexural, and shear strength along with energy absorption capacity, indicating suitability for protective structural applications (Ashok *et al.* 2024). A biodegradable hybrid composite reinforced with luffa fiber, Morinda tinctoria wood powder, and myrobalan seed powder was developed to improve mechanical and thermal stability (Kumar *et al.* 2023). Epoxy composites reinforced with casuarina fiber and nanoparticles (ZnO and ZrO₂) were developed to enhance mechanical and tribological performance. The ZnO-filled composite showed the best results with 85 MPa tensile strength, 125 MPa flexural strength, 7.5 kJ/m² impact strength, and 92 Shore D hardness (Barve *et al.* 2025).

Eco-friendly composites reinforced with Luffa cylindrica mats were fabricated using castor oil-based polyurethane to develop sustainable panels for civil construction. The composite significantly improved flexural strength and modulus compared with the neat matrix and showed potential as an alternative material for oriented strand board panels (Neves *et al.* 2022). Biodegradable polymer composites consisting of pectin, glycerol, and luffa fiber were synthesized by solvent casting for sustainable packaging applications. The optimized composition containing 15% luffa fiber exhibited improved tensile strength, good thermal stability up to 260 °C, and complete biodegradation within 75 days (Lal *et al.* 2023).

Polymer composites reinforced with diss, sisal, and luffa fibers were investigated to analyze the influence of fiber filling density on bending properties (Saada *et al.* 2024). Luffa fibers treated with 1% NaOH (90 min, 50 °C) showed improved properties, where tensile strength increased from 32.46 MPa to 84.26 MPa (159.58%), crystallinity increased by 43.7%, and thermal degradation mass loss decreased from 68% to 62% (Kharrati *et al.* 2026). Hybrid epoxy composites reinforced with treated luffa fibers and silane-modified cenospheres showed enhanced impact behavior. The optimized sample achieved a peak force of 1103.44 N and 37% higher energy absorption than the untreated composite due to improved fiber–matrix bonding (Gurjar *et al.* 2026). Hybrid kenaf/carbon/epoxy and kenaf/glass/epoxy composites fabricated by vacuum assisted resin infusion (VARI) showed major mechanical improvements over pure kenaf composites. The kenaf/carbon hybrid exhibited 529% higher tensile strength, 512% higher flexural strength, and 272% higher interlaminar shear strength (ILSS), outperforming

kenaf/glass hybrids despite a lower fiber volume fraction (Malik *et al.* 2022).

Hybrid laminates combining jute (J) and carbon (C) fibers were fabricated by VARI to improve durability and water resistance. The CJC laminate achieved a tensile strength of 377.1 MPa and a flexural strength of 405.4 MPa, while water absorption reduced to 5.69% compared with 9.3% for pure jute composites (Massou *et al.* 2025). A bio-epoxy composite reinforced with sisal and banana stem fibers was fabricated using vacuum infusion to develop eco-friendly structural materials. The composite exhibited density of 1041 kg/m³, flexural strength of 160.43 MPa, and Young's modulus of 2.17 GPa, demonstrating potential to replace conventional polymers (Ramón *et al.* 2023). Hybrid nano-silica/graphene reinforced jute–epoxy composites showed improved multifunctional properties. Tensile strength increased from 45.2 MPa to 93.8 MPa (~107%), flexural strength improved by 46.7%, thermal stability increased from 290 °C to 355 °C, and water absorption decreased by over 50% (Satish Kumar *et al.* 2026).

Hybrid kenaf–flax epoxy composites fabricated by VARI showed improved mechanical properties depending on stacking sequence. The FK₂ configuration achieved tensile strength of 59.8 MPa, while FK₃ showed the highest ILSS of 12.5 MPa and fracture toughness of 3302.3 J/m² (Malik *et al.* 2023). Hybrid jute/carbon fiber epoxy composites fabricated by VARI were evaluated for stacking sequence effects. The J₃C₄J₃ configuration achieved the highest tensile strength of 571 MPa, while C₂J₆C₂ exhibited flexural strength of 455 MPa, impact strength of 30 kJ/m², and the lowest water absorption of 3.8%, indicating suitability for lightweight structural applications (Abu Shaïd *et al.* 2020). Carbon fiber reinforced polymer composites modified with TiO₂ and SiC nanomaterials were fabricated using the vacuum-assisted resin transfer molding (VARTM) process to evaluate their mechanical and thermal performance. The composite containing 2 wt.% nanomaterials exhibited improved properties, with tensile strength, flexural strength, impact strength, hardness, and HDT increasing by 17%, 39%, 32%, 14%, and 21%, respectively, compared with the composite without nanomaterials (Modugu *et al.* 2022).

Hybrid PLA nanobiocomposites reinforced with 15 wt.% kenaf fiber, 15 wt.% aloe vera fiber, and 1 wt.% montmorillonite (MMT) clay were fabricated to evaluate mechanical and thermal behavior (Ramesh *et al.* 2020a). Poly lactic acid biocomposites reinforced with 30 wt.% treated aloe vera fiber and 0–3 wt.% MMT nanoclay were developed to study mechanical and barrier properties. The composite with 1 wt.% MMT exhibited the mechanical improvements of 5.72% in tensile strength, 6.08% in flexural strength, and 10.43% in impact strength, while the decomposition temperature increased

from 295°C to 299°C (T10) and 338°C to 350°C (T75) (Ramesh *et al.* 2020b).

In this study, ZrO₂ along with inorganic nanoparticles such as MMT, TiO₂, and SiC were incorporated to investigate their influence on mechanical efficiency of luffa fiber-reinforced EVO composites. The objective of investigation is to promote utilization of luffa fiber as promising reinforcement material for non-structural applications at construction and automotive sectors. In particular, these composites are considered suitable for lightweight interior components such as storage cabinets and panel structures used in aircraft cabin interiors, where reduced weight, sustainability, and adequate mechanical strength are essential requirements.

2. MATERIALS AND METHODOLOGY

2.1 Materials

Three-ply yarn composed of luffa (*Luffa cylindrica*) fibers were procured from Go Green Products and subsequently woven into plain-weave fabric structure with an areal density of 200 GSM. These standardized characterization methods ensured reliable evaluation of mechanical and physical characteristics of the luffa fiber yarn used as reinforcement in the composite fabrication process.

Zirconium oxychloride octahydrate (ZrOCl₂·8H₂O) supplied by Matrics Enterprises was used as the primary precursor for the synthesis of ZrO₂ nanoparticles. Sodium hydroxide (>97% purity) was employed as the precipitating agent to facilitate the formation of zirconium hydroxide during the co-precipitation process.

Natural MMT clay was used as the precursor material for the preparation of nano-montmorillonite. The raw clay was acid-activated using hydrochloric acid to remove impurities and increase surface activity, followed by drying and ultrasonic exfoliation to obtain nano-sized particles. Owing to its layered silicate structure and high surface area, nano-MMT acts as an effective reinforcing filler that improves the mechanical strength, thermal stability, and barrier properties of polymer composites.

Titanium isopropoxide (TTIP) supplied by Matrics Enterprises was used as the titanium precursor for the synthesis of TiO₂ nanoparticles.

Silicon dioxide powder (SiO₂, >97% purity) procured from Sigma-Aldrich was used as the precursor for the synthesis of silicon carbide nanoparticles.

2.2 Nanoparticle Preparation

Silicon carbide nanoparticles were synthesized using a carbothermal reduction technique. In this process,

a mixture of silica (SiO₂) powder and carbon black was thoroughly blended in a stoichiometric ratio and placed in a high-temperature furnace. The mixture was heated at temperatures between 1400–1600°C under an inert atmosphere (argon) for several hours to promote the reduction reaction. During the process, silica reacts with carbon to form SiC nanoparticles and carbon monoxide gas. The obtained product was cooled to room temperature, washed with diluted hydrochloric acid to remove impurities, rinsed with deionized water, and finally dried at 100°C to obtain fine SiC nanopowder.

Titanium dioxide nanoparticles were prepared using a sol–gel method. Titanium isopropoxide were used as the titanium precursor and dissolved in ethanol under continuous stirring. A mixture of distilled water and nitric acid was slowly added dropwise to initiate hydrolysis and condensation reactions. The solution was stirred for several hours until a homogeneous gel was formed. The gel was aged for 24 hours, dried at 80–100°C, and calcined at 450–500°C for several hours to obtain crystalline TiO₂ nanoparticles with improved purity and structural stability.

Nano-montmorillonite clay was obtained through acid activation and ultrasonic exfoliation of natural montmorillonite. Initially, raw MMT clay was dispersed in distilled water and treated with 1 M hydrochloric acid under constant stirring at 70–80°C for several hours to remove impurities and enhance surface activity. The treated clay were washed repeatedly with DI water until neutral pH were reached and then dried at 80°C. The dried clay was ground as fine powder and subjected to ultrasonic treatment to exfoliate the layered structure, producing nano-sized montmorillonite particles with improved dispersion characteristics.

Zirconium oxide nanoparticles was synthesized by co-precipitation method. ZrOCl₂·8H₂O was dissolved in DI water and stirred continuously to form clear solution. NaOH solution was then added dropwise under vigorous stirring until the pH reached approximately 10, resulting at formation of zirconium hydroxide precipitates. The precipitate was aged for several hours, filtered, and washed repeatedly with deionized water to remove residual ions. The collected product was dried at 100°C and calcined at 500–600 °C to obtain crystalline ZrO₂ nanoparticles.

2.3 Composite Fabrication

In this study, luffa fabrics were thoroughly rinsed with DI water to eliminate dust and other surface impurities while preserving the natural composition of the fibers. The cleaned fabrics were then dried at 35°C for 48hours, followed by desiccation for an additional 24hours to ensure complete removal of residual moisture (Dogan *et al.* 2025). After the drying process, layers of luffa fabric were manually arranged and impregnated by

EVO resin using hybrid fabrication technique that integrates vacuum-assisted resin infusion with autoclave processing. The VARI technique facilitated effective resin infiltration into the fiber network, while the autoclave was employed to regulate the curing conditions under controlled pressure at 40°C for 2 hours, ensuring proper consolidation and curing of composite laminates (Franco-Urquiza and Rentería-Rodríguez, 2021).

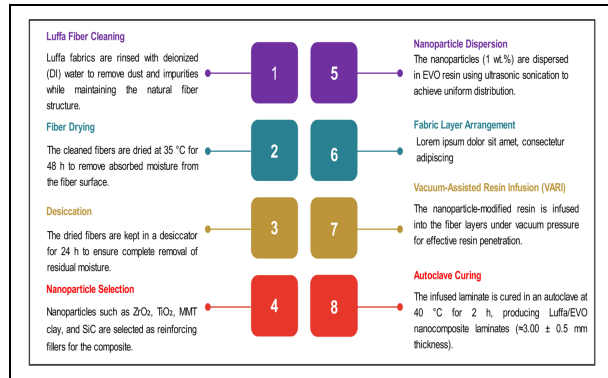


Fig. 1: Nanoparticle-filled luffa epoxy composite fabrication

Luffa/EVO (LE) bio-composites were reinforced with 1 wt.%, namely ZrO₂, TiO₂, montmorillonite (MMT), and silicon carbide (SiC). Prior to the impregnation stage, the nanoparticles were uniformly dispersed in EVO resin through an ultrasonic dispersion process by Q700 sonicator to enhance particle distribution and minimize agglomeration. Following dispersion, the nanoparticle-modified resin was infused into the luffa fiber layers during composite fabrication (Arunkumar *et al.* 2025). As a result, bio-composite laminates with average thickness of approximately 3.00 ± 0.5 mm were successfully produced (Fig. 1).

2.4 Characterization

The chemical structure of the LE bio-composites reinforced with different nanoparticles was examined using Fourier Transform Infrared Spectroscopy (FTIR) with a Bruker Alpha II spectrometer equipped with an attenuated total reflectance (ATR) module. The composite samples were directly positioned on ATR crystal surface for spectral acquisition. The spectra were collected in the wavenumber region 4000–400 cm⁻¹ (32 cumulative scans and spectral resolution of 4 cm⁻¹) to identify functional groups and interactions between the matrix and nanoparticles.

The crystalline characteristics of the nanoparticles were determined using X-ray diffraction (XRD) analysis carried out with a Bruker D8 Advance diffractometer employing Cu K α radiation. Diffraction was recorded over 10–80° at scan of 2°/min, enabling identification of crystalline phases and structural features of nanoparticles.

Viscoelastic properties of neat LE composite and LE nanocomposites were investigated by Dynamic Mechanical Analysis performed on Netzsch DMA 242 E Artemis instrument. Rectangular specimens measuring 50 mm × 10 mm × 3 mm were tested according to ASTM D7028. The storage modulus (E') and loss factor (tan δ) were measured in single cantilever mode while the temperature was increased from 30°C to 130°C at a heating rate of 3°C/min.

To determine the tensile behavior, unidirectional tensile tests were conducted in accordance with ASTM D3039 using a Zwick/Roell Z100 universal testing machine equipped with a 50 kN load cell. Composite specimens approximate dimensions of 250 × 25 × 3 mm were subjected to loading crosshead speed of 2 mm/min. For statistical reliability, eight samples from the same laminate panel were tested. Stress–strain response were recorded until fracture, and final mechanical properties were reported as mean values with standard deviation.

Strain measurements during the tensile test were captured using a non-contact optical extensometer (Zwick videoXtens system) equipped with a high-resolution camera and 30 mm lens. The system tracks surface markers on the specimen to accurately calculate strain, and the data were processed using the testXpert software platform. Additionally, X-ray computed tomography was employed as non-destructive method to inspect the internal structure and void distribution of the composites. The scanning process was performed using a Nikon XT H 225 micro-CT system capable of producing high-resolution three-dimensional images. Composite samples with dimensions of 80 × 6 × 3 mm were scanned to identify manufacturing defects, porosity levels, and internal structural features of the LE nanocomposites.

The surface morphology and fracture characteristics of the composites were analyzed using Scanning Electron Microscopy (SEM) performed on ZEISS Gemini SEM instrument at an accelerating voltage of 10–15 kV, with a working distance of 8–10 mm and a beam current of ~1 Na. These parameters allowed detailed examination of features such as fiber pull-out, particle agglomeration, interfacial adhesion, and micro-void formation, which play significant role at determining mechanical performance of composites.

3. RESULTS AND DISCUSSION

3.1 Microstructural Characterization

FTIR analysis was carried out to qualitatively identify the chemical characteristics and possible interactions of synthesized and heat-treated nanoparticles. The method was employed to determine surface functional groups of all nanoparticle and to evaluate their potential for improving interfacial interaction with the polymer matrix. Such information is

valuable for interpreting the mechanical behavior of the composites and understanding other phenomena influencing their overall performance. The FTIR spectra of ZrO_2 , MMT, TiO_2 , and SiC nanoparticles are presented in Fig. 2. In the ZrO_2 spectrum shown at bottom of Fig. 2, a broad peak around 3295 cm^{-1} corresponds to stretching vibration of N–H groups associated with amines, while peaks near 2920 and 2848 cm^{-1} indicate C–H stretching vibrations. The peak observed at approximately 1700 cm^{-1} were attributed to C=C vibrations, and the peak near 1589 cm^{-1} corresponds to NH_2 functional group. Additionally, the peak around 1503 cm^{-1} was related to the secondary amine groups. The weak peaks near 1656 and 1508 cm^{-1} were associated with C–OH and C–O stretching vibrations. A strong peak at approximately 1196 cm^{-1} was attributed C–O stretching vibration, while a weak peak around 936 cm^{-1} to C–H bending vibration. The presence of these groups indicates surface chemical activity that can promote stronger interfacial bonding with polymer chains (Ouaar *et al.* 2025).

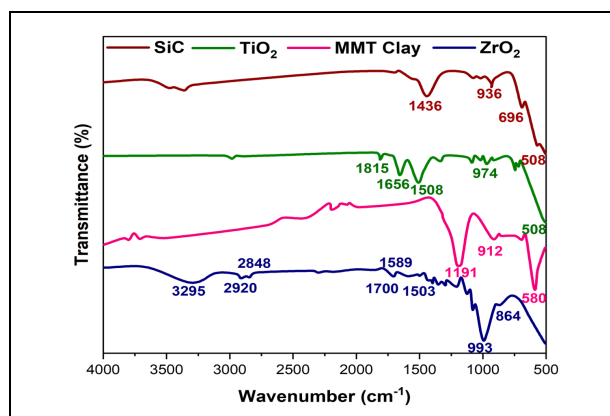


Fig. 2: FTIR spectra of reinforcing nanoparticles

The FTIR spectrum of the MMT particles exhibits three prominent absorption bands at 1191 , 912 , and 508 cm^{-1} , which are attributed Al–O stretching vibration, Si–O bonding and Si–O symmetric stretching vibration, respectively. Absence of characteristic peaks in the region of $3500\text{--}3700\text{ cm}^{-1}$, which are typically associated with hydroxyl groups in kaolin, indicates that heat treatment process were effective in transforming the material into calcined kaolin. This confirms the successful thermal modification of the particles and suggests improved structural stability suitable for composite reinforcement applications.

For TiO_2 nanoparticles, the FTIR spectrum displays a peak at 1815 cm^{-1} corresponding to C=O stretching vibrations. The peak at 1656 cm^{-1} is associated with N–H stretching, while the peak around 1508 cm^{-1} indicates C–H vibrations originating from acetate groups. Additional peaks near 974 cm^{-1} and 508 cm^{-1} correspond to hydroxyl-related vibrations and Ti–O bonding, confirming the formation of TiO_2 nanoparticles. The presence of surface –OH groups suggests potential active sites that can facilitate improved interfacial bonding with polymer chains.

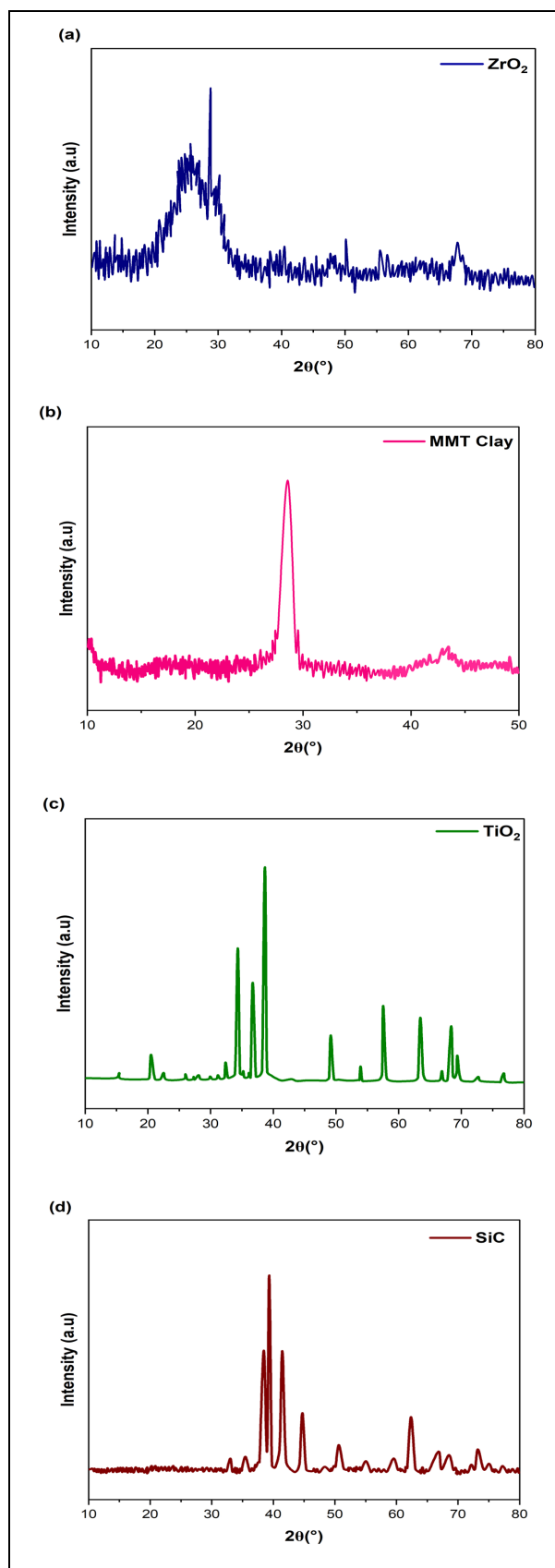


Fig. 3: X-ray diffraction pattern of (a) Zirconia (ZrO_2) (b) Montmorillonite (MMT), (c) Titanium dioxide (TiO_2) and (d) Silicon carbide (SiC)

The FTIR of SiC nanoparticles shows weak absorption peak near 1436 cm^{-1} , which is associated with O–H stretching vibrations. Peaks observed at 936 cm^{-1} and 696 cm^{-1} correspond to vibrational modes of $-\text{NO}_3$ and $-\text{HCO}_3$ groups, likely originating as residual by-products from the synthesis process. Furthermore, characteristic Si–C peaks were also observed, confirming the successful formation and presence of silicon carbide nanoparticles.

X-ray diffraction (XRD) is an effective technique used to examine the crystalline structure and possible interlayer modifications in nanoparticles (Senthil Kumar *et al.* 2024). Fig. 3 presents the XRD patterns of ZrO_2 , MMT, TiO_2 , and SiC nanoparticles. The diffraction pattern of ZrO_2 is illustrated in Fig. 3(a). These nanoparticles exhibit a broad peak with moderate intensity and a relatively diffused maximum, indicating the presence of partially crystalline structures and confirming the successful formation of the nanoparticle phase.

Table 1 summarizes parameters derived from XRD patterns of ZrO_2 , MMT, TiO_2 , and SiC nanoparticles. While the position of diffraction peaks is typically used to determine interlayer spacing, the peak broadening provides important information regarding the crystallite domain size within the nanoparticle structure. These domains were separated longitudinally by structural discontinuities and laterally by grain boundaries. Consequently, calculated crystallite width shows out-of-plane crystallite dimension, which corresponds to stacking order and perpendicular thickness of layered crystalline structure.

Table 1. XRD analysis

Nanomaterials	Diffraction Angle (2θ)	Crystal Plane (hkl)	Peak Width	Crystallite Size	Average Crystallite Size
ZrO_2	24.2	-11	1.33	8.11	8.0 nm
	28.2	-111	1.21	7.82	
	31.5	-200	1.15	8.02	
	50.3	-220	0.82	8.41	
	68.2	-311	0.74	7.96	
MMT Clay	19.8	-1	0.52	42.1	$38 \pm 2\text{ nm}$
	26.6	-2	0.44	36.5	
	29.5	-3	0.38	35.2	
	41.8	-4	0.31	37.3	
TiO_2	25.3	-101	0.32	38.1	$39 \pm 1\text{ nm}$
	37.8	-4	0.3	39.4	
	48	-200	0.29	40.2	
	54	-105	0.27	39.1	
	62.7	-204	0.25	38.7	
SiC	68.8	-116	0.24	39.3	$18 \pm 2\text{ nm}$
	35.6	-111	0.28	17.2	
	41.4	-200	0.27	18.4	
	60	-220	0.23	19.1	
	71.8	-311	0.22	17.6	
	75.5	-222	0.21	18.3	

X-ray diffraction (XRD) analysis of crystalline powders provides a reliable approach for estimating the average single-crystalline nanoparticles. Crystallite size (D) was determined from broadening of diffraction peaks, specifically full width at half maximum, observed in XRD patterns. This estimation is commonly performed using the Debye–Scherrer equation, which relates peak broadening to the average crystallite dimension.

$$D = \frac{K\lambda}{\beta \cos \theta} = \frac{0.89\lambda}{\beta \cos \theta} \quad \dots (1)$$

In this equation, FWHM represents full width half maximum of diffraction peak expressed as radians, while θ denotes Bragg angle. λ were X-ray wavelength, and K were factor constant, typically 0.89 for spherical crystals and about 0.943 for particles with cubic geometry.

Based on Equation (1), the FWHM value obtained for ZrO_2 nanoparticles was 1.27, and crystallite size estimated by Debye–Scherrer eqn was approximately 1.28 nm. Instead, broad diffraction between $2\theta = 25^\circ$ and 30° suggests inclusion of amorphous quartz-like phase characteristic of MMT. Using Debye–Scherrer equation, average crystallite size of MMT was calculated to be 8.01 nm. The XRD of TiO_2 nanoparticles (Fig. 3(c)) exhibits well-defined diffraction peaks at 2θ values of 21.3° , 35.3° , 38.4° , 58.4° , 39.3° , 41.5° , 44.7° , 50.7° , 62.3° , 66.7° , 68.5° and 73.2° . These reflections correspond to a hexagonal wurtzite-like crystal structure of TiO_2 with lattice parameters $\text{\AA}$ and $c = 39.21\text{ \AA}$. According to Eqn (1), calculated crystallite size of TiO_2 nanoparticles is approximately 1.2 nm. Fig. 3(d) presents XRD pattern of SiC nanoparticles, where diffraction observed at 2θ values of 35.7° , 41.4° , 60.0° , 71.7° and 75.4° indicate that SiC crystallizes in a monoclinic structure belonging to the C_2/c group ($a = 18.42$). In addition, the XRD pattern also corresponds to monoclinic phase associated with tenorite structure, as confirmed by diffraction peaks.

3.2 Viscoelastic Properties

Dynamic mechanical analysis (DMA) provides significant insight into viscoelastic properties of polymeric materials and their thermal transition behavior (Karim *et al.* 2024). The storage modulus represents amount of mechanical energy stored at material under deformation, whereas the damping factor reflects the extent of energy dissipation within the system. The DMA results, including damping factor and storage modulus curves, are illustrated in Fig. 4. Typically, storage modulus profile of thermoset polymers can be divided into three regions: glassy state, rubbery plateau, and glass transition region. The storage modulus at glassy region changes depending on type of nanoparticle incorporated, as depicted in Fig. 4(a). The storage modulus values for LE– ZrO_2 and LE–MMT composites remain relatively stable before reaching the onset of glass transition

temperature (T_g). Conversely, LE-TiO₂ and LE-SiC composites exhibit a gradual decline in storage modulus with increasing temperature, resembling the behavior observed in the unfilled KE composite. For all composite samples, a significant reduction in stiffness occurs within the glass transition region due to increased segmental mobility of the polymer chains, eventually leading to plateau region.

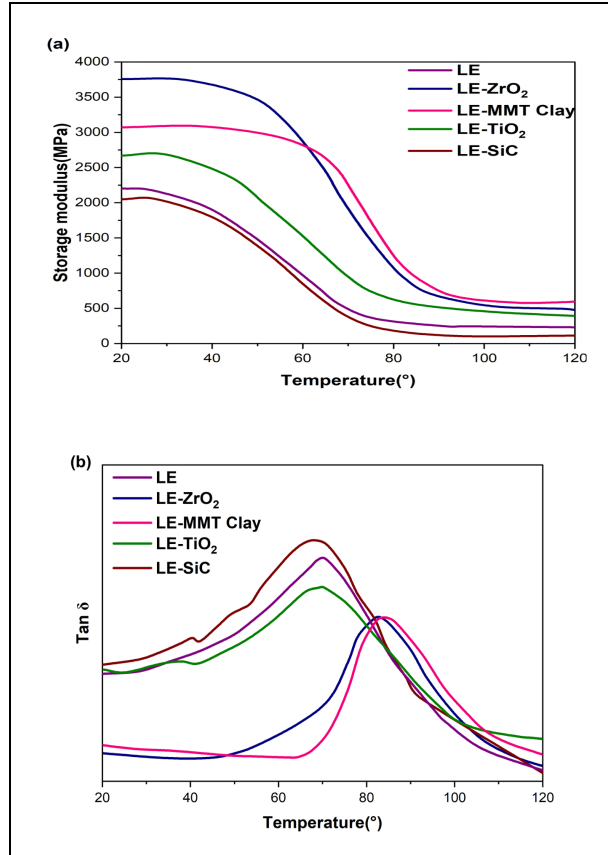


Fig. 4: DMA results of LE (a) Storage modulus variation and (b) Damping factor ($\tan \delta$)

Storage modulus of LE composite in glassy region were measured at 2200 MPa. The incorporation of nanoparticles increased the modulus to 3750, 3050, and 2650 MPa for LE-ZrO₂, LE-MMT, and LE-TiO₂ composites, respectively. In contrast, KE-SiC exhibited the lowest storage modulus value of 2050 MPa. These results indicate that rigid nanoparticles effectively enhance the stiffness of the composite by acting as secondary reinforcement within the EVO matrix. However, SiC nanoparticles tend to behave more like plasticizing agents, contributing to load distribution without significantly improving stiffness. This observation is consistent with previous studies, which reported that the morphology and relatively low concentration of nanoparticles influence the reinforcement mechanism in composite materials (Ashok and Kani, 2022).

Stiffness observed at rubbery plateau region varies depending on the type of nanoparticles and were closely related to density of composite system. The unfilled LE composite exhibits storage modulus of about 230MPa approximately at 100°C. At the same temperature, storage modulus values for LE-ZrO₂, LE-MMT, and LE-TiO₂ increase to 540, 590, and 450 MPa, corresponding to improvements of 135%, 157%, and 96%.

$$\rho = \frac{E_r'}{3RT_r} \quad \dots (2)$$

Based on calculated ρ values (eqn 2) listed at Table 2, it can be observed that stiffness at rubbery plateau raises notably with rise in physical crosslinking within the composite system. This behavior is attributed to inclusion of nanoparticles with large surface areas, like TiO₂, along with high- ratio fillers like clay, which function effective physical crosslinking sites and restrict localized molecular mobility of the polymer chains. In contrast, the KE-SiC composite exhibits the lowest crosslinking density among the prepared samples.

Table 2. Viscoelastic properties of LE nanocomposites

Material Composition	Glassy Storage Modulus (MPa)	Rubbery Plateau Modulus (MPa)	Crosslink Density (mol/cm ³)
LE	2200	230	2.30×10^{-1}
LE- ZrO ₂	3750	540	5.40×10^{-1}
LE-MMT	3050	590	5.90×10^{-1}
LE-TiO ₂	2650	450	4.50×10^{-1}
LE-SiC	2050	100	1.00×10^{-1}

Fig. 4(b) indicates that incorporation of SiC and TiO₂ does not modify position of $\tan \delta$ peaks, which appear around 70 °C. For unfilled LE composite, $\tan \delta$ peak is observed at approximately 70 °C, indicating that glass transition temperature remains nearly unchanged with addition of these nanoparticles. In contrast, higher T_g values with maximum peaks were observed at 82 °C and 84 °C for LE-ZrO₂ and LE-MMT composites, respectively. This positive shift at $\tan \delta$ position were provided to enhanced interfacial interactions among reinforcements and polymer matrix, which restrict segmental mobility of polymer chains at LE bio-composites.

3.3 Mechanical Performance of the Composites

The stress-strain curves for unfilled LE composite and the corresponding LE-based nanocomposites are presented in Fig. 5.

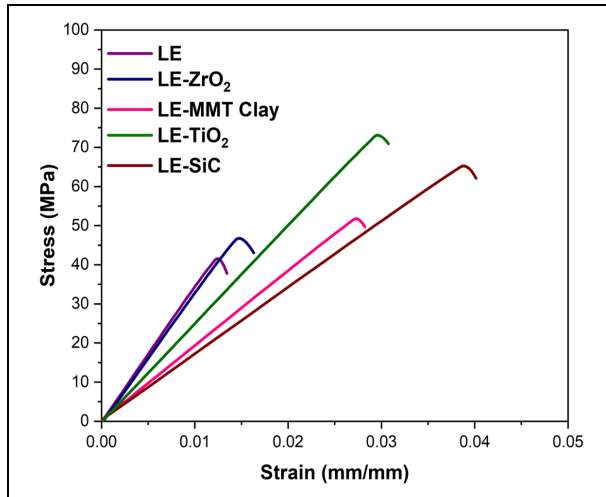


Fig. 5: Stress–strain behavior of LE nanocomposites

Initially, stress–strain curves exhibit a linear elastic response. However, a closer examination reveals a slight variation in slope at unidirectional tensile loading. Initial linear region corresponds to elastic deformation of composites. The subsequent non-linear region is associated with stick–slip mechanism commonly observed in woven composite structures. In this stage, the materials display gradual elastoplastic deformation prior to abrupt failure. Among the tested samples, the LE–SiC composite showed a completely linear elastic response without noticeable non-linear behavior.

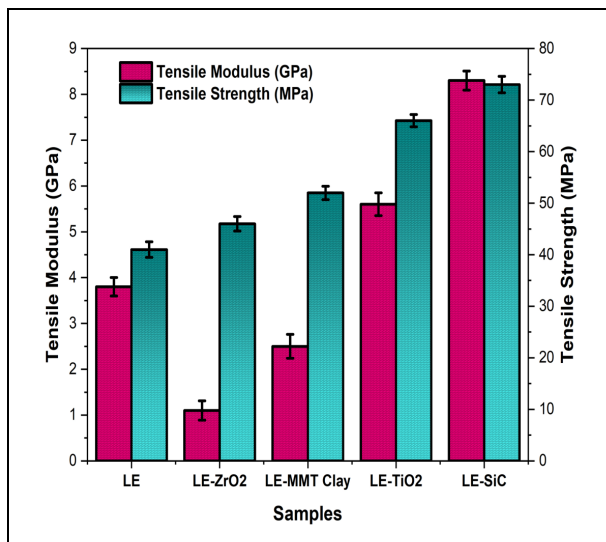


Fig. 6: Uniaxial tensile properties of LE composites

With respect to YM, a decrease in stiffness is observed when TiO_2 and SiC nanoparticles were incorporated into LE bio-composites. Conversely, a significant improvement in stiffness is noted for composites reinforced by ZrO_2 and MMT nanoparticles. Meanwhile, the relatively non-rigid nature of SiC particles contributes to a less tough response in the LE

bio-composites. Additionally, the nearly spherical morphology of SiC nanoparticles can reduce stiffness through a “ball-bearing” effect, which facilitates localized movement and is also associated with the lower crosslinking density determined from the DMA analysis in this study.

In contrast, ZrO_2 and MMT nanoparticles possess various morphologies compared with TiO_2 and SiC. These materials exhibit structures that can undergo exfoliation or interlayer sliding. The well-dispersed platelets, characterized by low percolation thresholds and high aspect ratios, contribute to enhanced stiffness in LE bio-composites. This improvement occurs because the intercalated platelet structures create constrained regions that restrict mobility of polymer chains. Additionally, surface platelets of ZrO_2 and MMT promote closer packing of polymer chains, as the thin layered configuration can fold and adhere strongly to the matrix. This compaction effect strengthens the composite, resulting in improved mechanical strength and reduced strain prior to failure.

Incorporation of small quantities of nanofillers into fiber-reinforced polymer can significantly enhance not only physical and chemical characteristics but also the fracture and mechanical performance, making them suitable for structural applications. As illustrated in Fig. 6, strength of LE bio-composites increases notably with the addition of various nanoparticles. The tensile strength of unfilled LE composite is 41 MPa, which gradually increases with the incorporation of SiC and TiO_2 nanoparticles, which possess rock-like morphologies, resulting in strength improvements of approximately 43 % and 37%, respectively. In contrast, the introduction of layered nanoparticles such as MMT and ZrO_2 leads to more pronounced enhancements, increasing the tensile strength by nearly 21.15% and 37.87%. Although TiO_2 and SiC nanoparticles generally exhibit predominantly spherical morphology that promotes crack due to localized stress concentrations around the particles, the observed improvement in strength can be attributed to effective dispersion and surface modification of nanoparticles.

Failure analysis was performed according to D3039M / ASTM D3039 and the fracture modes were classified accordingly. The LE biolaminate exhibited a fracture type AIT, whereas LE– ZrO_2 , LE–MMT, and LE– TiO_2 composites showed fracture type AGM, and the LE–SiC composite displayed fracture type AGB. In all specimens, angled fracture patterns were observed, which can be attributed to slight misalignment in the woven fabric structure, leading to non-uniform stress distribution across the transverse section. Additionally, the warp and weft yarns did not exhibit perfect symmetry during tensile loading, likely due to the handcrafted weaving process, which may result in irregular failure behavior. Nevertheless, no evidence of particle

agglomeration or bubble-related defects was detected during the fracture examination.

Voids or air pockets may develop because of incomplete resin infiltration, trapped air, or interruptions in resin flow resulting from inclusion of nanoparticles within epoxy matrix. Additionally, these voids vary in shape, size, and orientation depending on intrinsic characteristics of composite system as well as processing conditions like temperature and pressure during fabrication. X-ray computed tomography has become increasingly valuable non-destructive evaluation technique for fiber-reinforced polymer (FRP) composites, as it utilizes differences in material density to identify and quantify defects such as voids, inclusions, or bubbles within the laminate structure. Moreover, this technique assists in determining the volume fraction of different constituents in the composite. Fig. 7 presents measured volume fractions of unfilled LE composite and the corresponding LE nanocomposites.

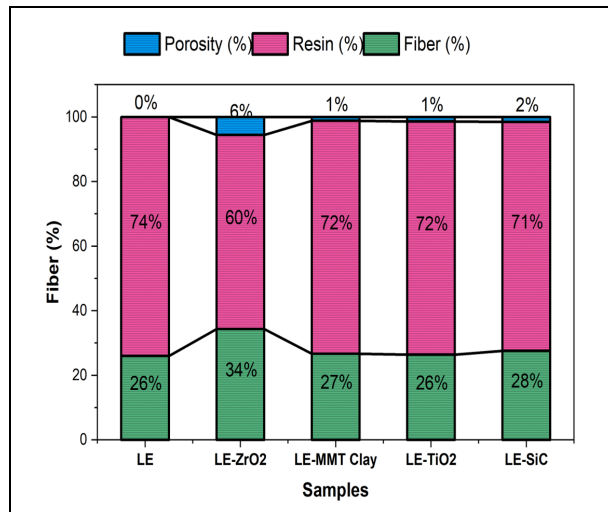


Fig. 7: Volume fraction histograms from CT analysis

The porosity content observed at LE-ZrO₂ composites was significantly higher compared to other LE nanocomposites investigated in this study. This behavior may be associated with particle agglomeration and insufficient dispersion of ZrO₂ sheets, which can modify overall volume fraction and negatively influence mechanical performance of composites. In contrast, LE-MMT composite exhibited a lower tendency for void formation, attributed to the exfoliated and uniformly dispersed clay that contributed to improved structural integrity and enhanced mechanical properties.

3.4 Scanning Electron Microscopy

Scanning electron microscopy (SEM) was used to examine fracture surface morphology and nanoparticle dispersion in LE composites, as shown in Fig. 8. Fig. 8(a) illustrates the unfilled LE composite, which exhibits a relatively smooth fracture surface with noticeable fiber

pull-out, micro-voids, and interfacial gaps among luffa fibers and epoxy matrix, indicating weak interfacial adhesion and poor stress transfer. Fig. 8(b) shows the LE-ZrO₂ composite, where dispersed ceramic particles are embedded within the matrix, producing a rougher fracture surface that suggests improved energy absorption, although minor particle agglomeration is still observed. Fig. 8(c) presents the LE-MMT composite, characterized by a compact morphology with layered platelet dispersion that enhances fiber-matrix bonding and restricts crack propagation, thereby reducing fiber pull-out. Fig. 8(d) depicts the LE-TiO₂ composite with spherical nanoparticles distributed within the matrix, acting as stress transfer sites between the fiber and matrix, though slight nanoparticle clustering appears in certain regions. Fig. 8(e) represents the LE-SiC composite, where embedded ceramic particles generate a rough and irregular fracture surface, indicating improved interfacial interaction, despite partial clustering caused by the high surface energy of SiC particles. The SEM analysis confirmed improved load transfer between fiber, filler, and matrix.

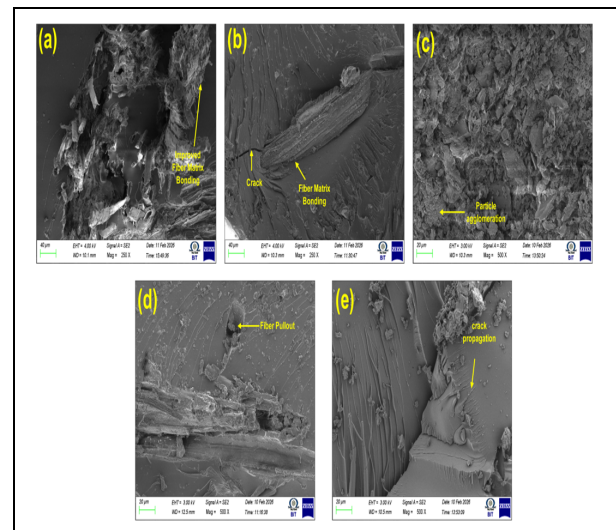


Fig. 8: SEM micrographs of LE composites: (a) LE composite, (b) LE-ZrO₂, (c) LE-MMT, (d) LE-TiO₂ and (e) LE-SiC

4. CONCLUSIONS

Luffa fiber-reinforced bio-composites incorporating various nanoparticles were successfully fabricated using a hybrid manufacturing approach in this study. The nanoparticles were synthesized and processed according to their specific characteristics. The FTIR analysis confirmed the presence of functional groups that facilitate improved interfacial interactions between the nanofillers and the polymer matrix. Structural characterization through XRD indicated that ZrO₂ exhibited limited structural connectivity, which contributed to particle agglomeration observed in tomography analysis. Additionally, thermal treatment of calcined kaolin resulted in the formation of an MMT

structure, while synthesis of metallic nanoparticles produced crystalline phases characteristic of TiO₂ and SiC nanoparticles.

Dynamic mechanical analysis showed that storage modulus at glassy area increased from about 2200MPa for the neat LE composite to approximately 3750MPa, 3050MPa, and 2650MPa for LE–ZrO₂, LE–MMT, and LE–TiO₂ nanocomposites, respectively. Similarly, in the rubbery plateau region at around 100 °C, the storage modulus increased from ~230MPa to about 540MPa, 590MPa, and 450MPa for LE–ZrO₂, LE–MMT, and LE–TiO₂ composites, respectively, indicating improved stiffness and stronger filler–matrix interactions. Furthermore, the glass transition temperature shifted from approximately 70°C for the neat LE composite to about 82°C and 84°C for LE–ZrO₂ and LE–MMT nanocomposites, confirming restricted polymer chain mobility due to enhanced interfacial bonding.

The layered morphology of ZrO₂ and MMT resulted in smaller crystallite sizes compared with metallic oxide nanoparticles, as calculated using the Debye–Scherrer equation. Particle morphology plays a significant role in influencing the mechanical and viscoelastic behavior of LE bio-composites. The larger surface area of ZrO₂ and MMT platelets contributed to a notable improvement in stiffness of LE bio-composites. Furthermore, the fillers evaluated in this study demonstrated effective load transfer capability within the composite structure, resulting in a significant enhancement in the overall strength of the biolaminate.

Computed tomography analysis revealed that the porosity level in LE–ZrO₂ composites was significantly higher than that observed in the other LE nanocomposites. This behavior is mainly attributed to particle agglomeration and inadequate dispersion of ZrO₂ sheets, which altered the volume fraction and consequently affected the mechanical performance of the composites. In contrast, LE–MMT composites exhibited a lower tendency for void formation due to the exfoliated and uniformly dispersed platelets, which contributed to improved structural integrity and enhanced mechanical properties.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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