

Effect of Nano-Silica Reinforcement in the Development of Bamboo Fiber Mat Epoxy/Palm Oil Biocomposites

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This research article describes the effects of nano-silica (nSiO₂) reinforcement in the development of epoxy/palm oil-filled bamboo fiber mat composites by characterising their physical and mechanical properties using different techniques as per ASTM standards.

Thirteen composites (S1 to S13) were fabricated with a different number of bamboo fiber mat layers (0, 2, 3, and 4) and varying weight percentages of nano-silica (0, 3, 5, and 7 wt%).

The dispersion of nSiO₂ into epoxy/palm oil resin was facilitated by mechanical mixing followed by a sonication technique. The nSiO₂/epoxy/palm oil was reinforced with bamboo fiber mats by hand lay-up method. The morphological features were examined by scanning electron microscopy. The properties of the epoxy/palm oil-based bamboo fiber mat/nano silica-reinforced composites indicated that the tensile strength of the composites improved with nSiO₂ wt.% upto 5 wt.% and decreased thereafter suggesting that the beneficial properties were observed up to 5 wt.% nSiO₂ content exhibiting superior thermal strength and resistance to temperature degradation compared to other nano silica biocomposites.

Keywords: NFRPC, polymer composite, natural fiber, sustainability, Mechanical strength, Thermal Stability, Bamboo fiber mat, Nano silica.

1. Introduction

The field of composite materials has seen significant growth in recent years, driven by the demand for materials with tailored properties across various engineering applications (Chen et al., 2020; Irez et al., 2020; Nayak et al., 2020). Composites, characterized by their heterogeneous nature, enable the synergistic combination of different materials to achieve desired performance characteristics. They are primarily classified into three categories based on their host materials: Metal matrix composites, Ceramic matrix composites, and Polymer matrix composites (Suresha et al., 2021). Among these, polymer matrix composites, particularly those based on thermoset resins like epoxy, have garnered considerable attention due to their versatility and adaptability. Epoxy, a widely studied thermoset polymer, offers several advantages, including ease of fabrication, faster curing times, chemical and flame

retardancy, and the ability to maintain structural integrity at elevated temperatures. Despite extensive research, epoxy-based composites continue to present numerous avenues for exploration in engineering applications. To enhance the properties of thermoset resins, various reinforcements are introduced into the matrix, including fibers (short fibers and long fiber woven fabrics), particulates, whiskers, and flakes. Recent interest has surged in natural fibers derived from sources such as pineapple, banana, flax, and jute (Lokantara et al., 2020; Parre et al., 2020; Prabhu et al., 2021; Subbiah et al., 2022; Suresh et al., 2020; Vinod et al., 2020). However, these fibers are still striving to fulfil the high strength-to-weight ratio demanded by engineering applications. Recent advancements have focused on incorporating nanoparticles into epoxy-based systems. Nanoparticles used in composites include allotropes of carbon (graphite, graphene, carbon nanotubes), metals and metal oxides (TiO₂, SiO₂, Al₂O₃, CaCO₃), ceramic nanoparticles (boron nitride, boron carbide, nanoclay), and nano-silica (nSiO₂), which has shown promise in enhancing the mechanical, tribological, and thermal properties of composites (Coetzee et al., 2020; Hariharan et al., 2023; Ogbonna et al., 2022; Omerović et al., 2021; Ren et al., 2020; Srinivasa Perumal et al., 2023)(Coetzee et al., 2020; Hariharan et al., 2023; Ogbonna et al., 2022; Omerović et al., 2021; Ren et al., 2020; Srinivasa Perumal et al., 2023; Suresha et al., 2021).

The present study investigates the potential of treated bamboo fiber mats as reinforcement in a novel composite system. Bamboo fibers represent an underutilized natural resource in polymer composites. Recent advancements in fiber treatment techniques have addressed limitations such as the tendency to form aggregates with polymer matrices and poor moisture resistance (Kaima et al., 2023; Mohamad et al., 2020). Pretreatment processes facilitate the production of bamboo fiber mats, offering improved handling and processing characteristics compared to loose fibers. The matrix material consists of a blend of epoxy resin, palm oil, and nano-silica, representing an innovative approach to composite formulation. Key aspects of the research include systematic variation of bamboo fiber mat layers (0, 1, 2, 3, and 4) within the epoxy-palm oil-nano-silica matrix, comprehensive evaluation of the impact of fiber content on overall composite properties, and assessment of mechanical, thermal, and morphological characteristics of the resulting composites.

This research seeks to contribute valuable insights to the field of natural fiber-reinforced polymer composites. By exploring the unique arrangement of treated bamboo fiber mats with a blended epoxy-palm oil-nano-silica matrix, the study aims to inform the development of sustainable, high-performance materials for applications in automotive, construction, and consumer goods industries. The findings have the potential to address the growing demand for environmentally friendly alternatives to traditional composite materials while maintaining or improving performance characteristics.

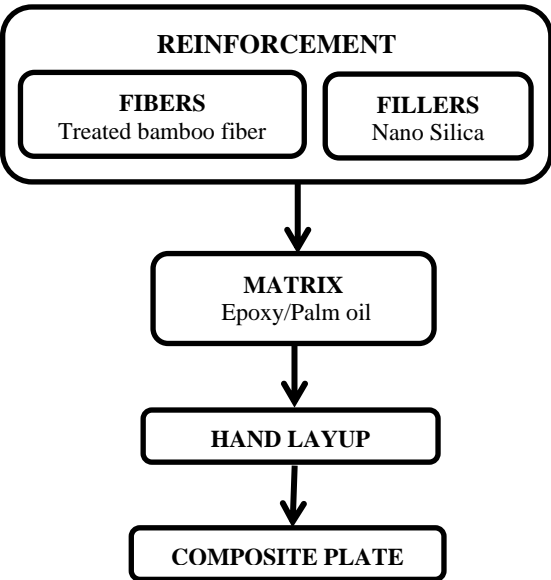


Figure 1Stages in Composite Preparation

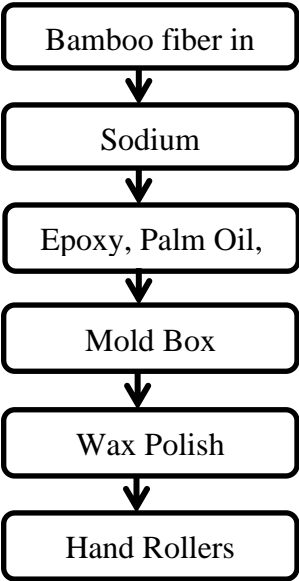


Figure 2Materials Used for Composite Preparation

2. Experimental Details

2.1 Materials and Methods

Araldite HY 951 epoxy resin and Aradur 22962 hardener procured from M/s Seenivasa Solutions Pondicherry, India were used in the composites fabrication. The density of the epoxy and hardener was 1.1–1.2 g cm⁻³ and 0.8–0.9 g cm⁻³ respectively.

The resin and hardener were mixed in 100:25 weight proportion as prescribed by the manufacturer. The bamboo fiber as mat with an areal mass 240 GSM and thickness 0.34 mm was procured from Metro Composites, Pvt. Ltd. India. Alkaline NaOH was acquired from Indian Scientific Solution, India. The nSiO₂ with dimension 10–20 nm was procured from Ultrananotech Private Limited, Bangalore, India. As per the manufacturer data sheet the density of powder nSiO₂ ranged from 2.2 to 2.6 g cm⁻³ at 25 °C. This work enumerates the materials used, experimental methods, and testing adopted for composite fabrication. Figure 1 presents the stages involved in preparing the composite and Figure 2 presents Materials Used for Composite Preparation.

2.2 Alkali treatment

The retted bamboo fiber mat was manually cleaned to remove any unwanted woody core. The process involved soaking the bamboo fiber mat in 5 wt % NaOH solutions for 1 day at ambient temperature. The fiber mat was additionally desiccated in an oven at 110°C for 24 hours (Diab et al., 2023). The fiber mats were deep rinsed in distilled water to remove alkali content from the fiber surface till it became neutral and scrubbed with pulp and paper fiber scrubber for approximately 45 minutes to eliminate chemical residues and to increase the surface area and decrease the hydrophilic groups which cause inadequate adhesion at the fiber mat/matrix interface (Mohammed et al., 2022).

2.3 Preparation of Composite

A 300 X 300 X 30 mm rectangular glass box was used as a mould, with wax applied as a releasing agent to prevent the adherence of the cured composites with the mould walls. The fabrication process began with the preparation of the epoxy-palm matrix, which is mixed steadily with the hardener for 10 minutes to ensure proper curing and quality. This mixture is then poured uniformly into the mould box. NaOH-treated bamboo fiber mats (10.5, 15.75 and 21 wt%) are layered sequentially, with each layer compacted using hand rollers to eliminate air bubbles. An 80kg weight is applied to compress the entire composite within the mould. After a 24-hour curing period, the composite was removed and subjected to an additional post-curing process for 24-hour in open air (Agwa et al., 2022; Karthikeyan & Naveen, 2024). This method is employed to produce composite samples S2, S6, and S10 without nano-silica particulate fillers. For the remaining samples, the resin mixture is modified by incorporating 3, 5, or 7 weight fraction of nano-silica particulate fillers, blended thoroughly before fiber mat reinforcement. The subsequent fabrication steps remain consistent across all samples. Table 1 presents the detailed composition of the composites. Once cured, the composites are extracted from the mould as plates (Figure 3) and subsequently cut into specimens suited to ASTM standard dimensions for testing.



Figure 1Bamboo fiber mats / nano silica particles reinforced with palm oil and epoxy biocomposites

Table 1Composition of bamboo fiber mat nano biocomposites

Composite Samples	Compositions				
	Epoxy Wt%	Palm oil Wt%	Bamboo fiber mat layers	Bamboo fiber mat Wt %	Nano silica (Wt%)
S1	70	30	0	0	0
S2	59.5	30	2	10.5	0
S3	59.5	27	2	10.5	3
S4	59.5	25	2	10.5	5
S5	59.5	23	2	10.5	7
S6	54.25	30	3	15.75	0
S7	54.25	27	3	15.75	3
S8	54.25	25	3	15.75	5
S9	54.25	23	3	15.75	7
S10	49	30	4	21	0
S11	49	27	4	21	3
S12	49	25	4	21	5
S13	49	23	4	21	7

3. Characterization

3.1 Tensile test (ASTM D638)

Tensile testing was conducted using Unitek-94100(2003) universal testing machine. Samples were prepared in accordance with ASTMD638 standard (Aykanat & Ermeýdan, 2022), measuring 175mm in length, 25mm in width, and 3mm in thickness. Five specimens were tested for each case, with the average value reported as the result. The testing parameters included a head displacement range of 5 to 250 mm/min and a load range of 0 to 100 kN for all samples. To quantify the materials ductility, the percentage elongation at break was calculated. This measures the extent to which a material can be stretched before failure. The initial gauge length L_0 and the ultimate length of the gauge section after fracture L_f were recorded.

The percentage elongation at break was then determined by

$$\text{Elongation at break (\%)} = ((L_f - L_o) / L_o) \times 100 \text{ ---- (1)}$$

Where L_f is the final length and L_o is the initial length.

3.2 Scanning electron microscopy (SEM)

Scanning electron microscopy was employed to investigate the interaction between the fiber and matrix, which influences the mechanical strength of the composite material. The fractured surfaces of the samples were prepared by cutting and uniformly coating with platinum prior to examination. SEM analysis was conducted using a JEOL JSM 6610 LV scanning electron microscope at an acceleration voltage of 20 kV. This technique allowed for detailed observation of the interfacial bonding characteristics, providing insights into the microstructural properties of the composite materials.

3.3 Thermogravimetric analysis (TGA)

Thermal properties of the hand-layup fabricated composites were evaluated using thermogravimetric analysis (TGA), following the ASTM E1868 standard. This quantifies the weight loss of a material as a function of temperature or time in a controlled atmosphere. The analysis was conducted using a NETZSCH STA 449F3 instrument at Annamalai University. Samples weighing 10 grams each were heated from 27 to 597 °C at a rate of 5 °C per minute in a nitrogen atmosphere. TGA assessed the thermal degradation and stability of the composites, with thermal stability being evaluated based on significant weight loss occurring at specific temperatures. This analysis provides the composites behavior under elevated temperatures, which is essential for understanding their potential applications and limitations in various thermal environments.

4. Results and Discussion

4.1 Tensile Test

The tensile strength of the biocomposites increased from 46.3 MPa for the control sample (S1) without bamboo fiber mat or nano silica to a maximum of 75.3 MPa for the triple-layer bamboo fiber mat with 5 wt% nano silica (S8). A linear increase in tensile strength was observed up to this optimal configuration, after which it began to decline with further reinforcement. Among the thirteen samples, those with 3 wt% nano silica and triple-layer bamboo fiber mat exhibited the highest tensile strengths of 69.6, 75.3, and 70.1 MPa, respectively. These results demonstrate the effectiveness of bamboo fiber mat layers and nano silica in enhancing the tensile strength of the biocomposite structure.

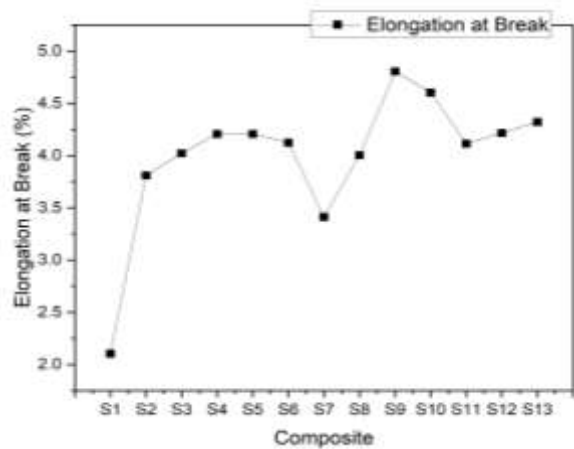


Figure 4. Tensile strength obtained for bamboo fiber mat nano biocomposites

The mechanical properties of these biocomposites are influenced by various factors, including matrix composition, fiber mat characteristics, resin type, nano particle properties, and fabrication processes(Kumar et al., 2023) . While increasing the number of fiber mat layers and nano particle content typically enhances tensile strength, our study revealed a decrease beyond the optimal configuration (triple-layer mat with 5 wt% nano silica)**Error! Reference source not found..** This decline is attributed to weak interfacial bonding and ineffective stress transfer due to inadequate wetting and agglomeration at higher reinforcement levels(Harikumar & Devaraju, 2021).

Percentage elongation at tensile strength reached a maximum of 4.8% for the S8 sample (**Error! Reference source not found.**), corresponding to the highest tensile strength. This property is crucial for understanding the material's ductility and flexibility in biocomposite applications(Chee et al., 2021).The optimal elongation is achieved through a balance between natural oil and polymer content, as well as fiber mat optimization.

These findings contribute to the development of bamboo fiber mat nano biocomposites with enhanced tensile properties for various engineering applications.

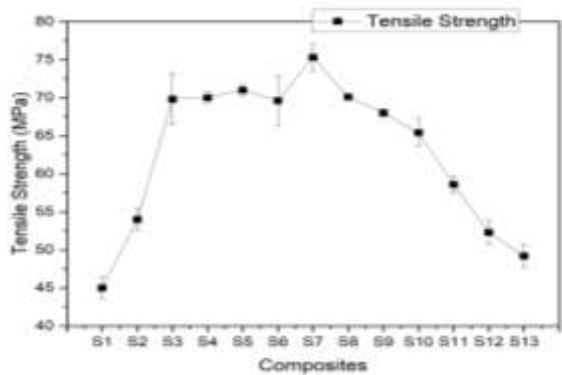
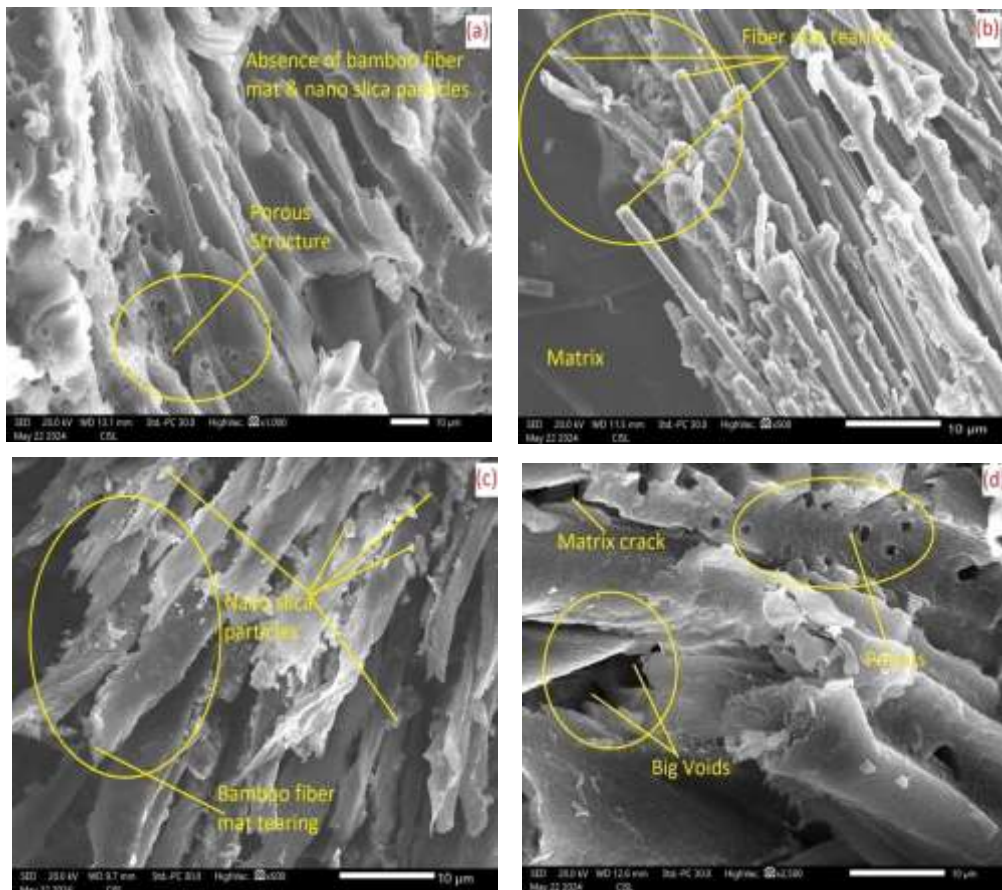


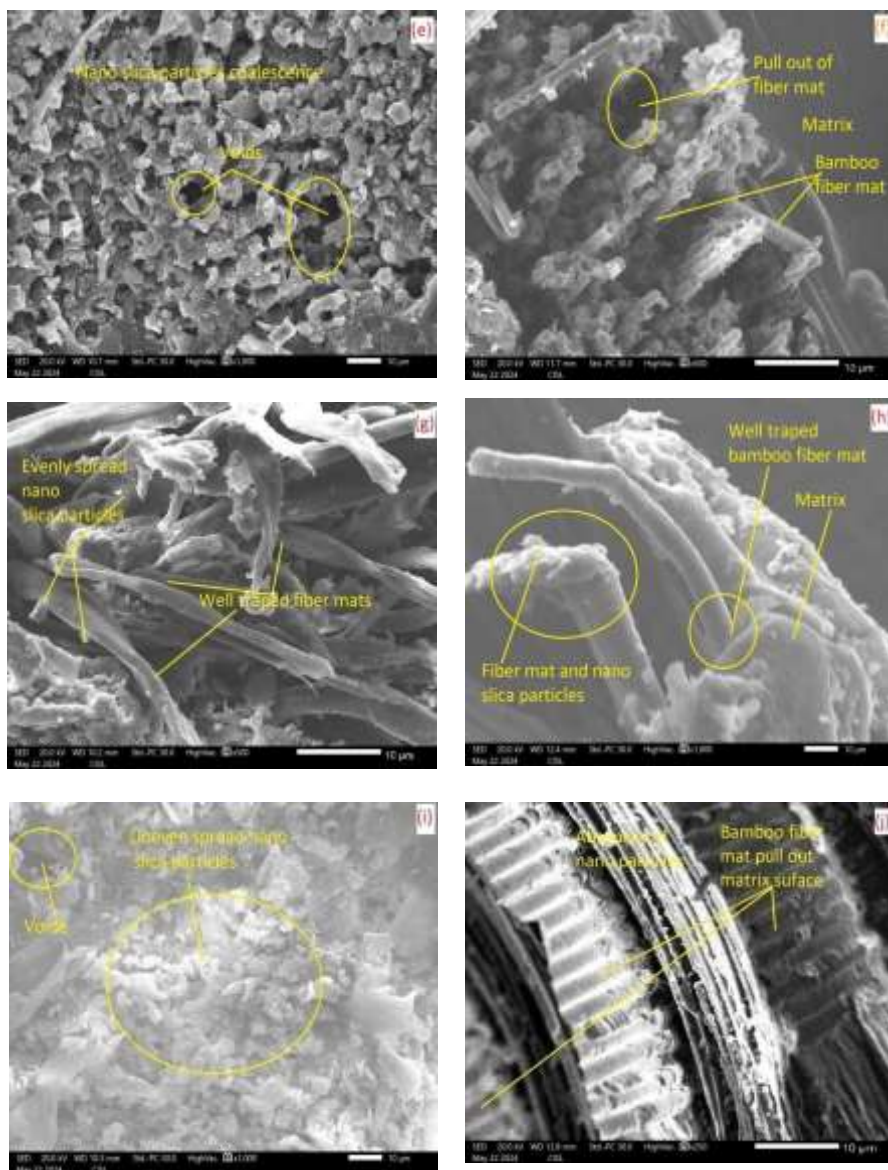
Figure 2. Tensile strength elongation obtained for bamboo fiber mat nano biocomposites

4.2 Scanning Electron Microscopy (SEM)

Scanning electron microscopy revealed the microstructural characteristics of bamboo fiber mat nano biocomposites with varying compositions. Sample (S1) without bamboo fiber mat or nano silica exhibited a highly porous structure (**Error! Reference source not found.** (a)). Two-layer biocomposites (S2-S5) with 0-7 t% nano silica showed voids, porosity, particle coalescence, and fiber mat tearing (**Error! Reference source not found.** (b,c,d,e)) indicating poor compatibility between components. This resulted in inferior mechanical properties compared to three- and four-layer biocomposites(V. Balaji et al., 2024).

Notably, triple-layer biocomposites (S6-S9) demonstrated superior microstructural features, including absence of cavities, well-trapped fiber mat, and adequate fiber mat-matrix adhesion (**Error! Reference source not found.** (f,g,h,i)). This correlated with the highest tensile strength (75.3 MPa) observed in the triple-layer sample with 5 wt% nano silica. Four-layer biocomposites (S9-S13) exhibited some uneven distribution, minor cracks, and small cavities (**Error! Reference source not found.** (j,k,l,m)) contributing to a slight decrease in tensile strength compared to triple-layer samples(Binoj et al., 2023).





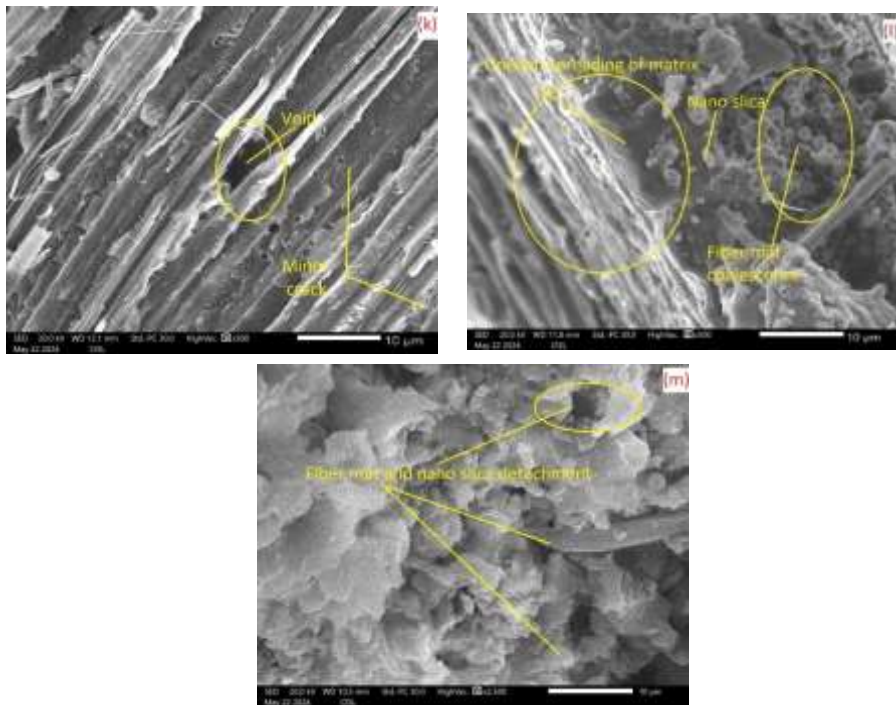


Figure 6 SEM Images of fractured Specimens

4.3 Thermogravimetric Analysis (TGA)

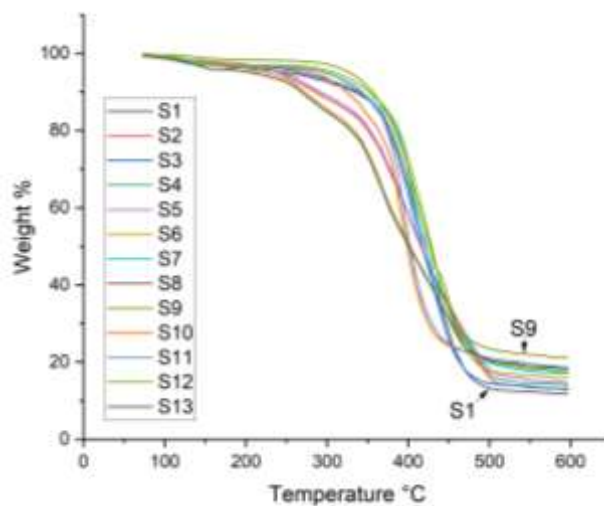


Figure 7TGA for bamboo fiber mat nano biocomposites

TGA(**Error! Reference source not found.**) revealed the thermal stability characteristics of the biocomposites (S1-S13).All samples demonstrated good thermal stability up to 230°C, with weight losses of only 2.5-8%. A 22% weight loss occurred between 290-350°C due to cellulose and

hemicellulose degradation. Major weight loss (350-482°C) was attributed to the decomposition of hemicelluloses, cellulose, and lignin.

Complete decomposition of combustible components and char formation occurred around 510°C. Total weight losses ranged from 79.4 to 88.2 wt%. Notably, the S9 sample (triple-layer bamboo fiber mat with 7 wt% nano silica) exhibited the highest thermal stability with the least weight loss (79.4 wt%)(A. Balaji et al., 2023).

These findings demonstrate the significant impact of bamboo fiber mat layering and nano silica content on both microstructural characteristics and thermal stability of the biocomposites.

5. Conclusion

This study investigated the properties of bamboo fiber mat nano biocomposites reinforced with nano silica particles yielding several significant findings. The three-layer bamboo fiber mat with 5 wt% nano silica (S8) demonstrated superior performance, achieving the highest tensile strength of 75.3 MPa among the thirteen tested biocomposites. SEM analysis of S8 revealed well-trapped fibers, the absence of cavities, and exceptional adhesion at the fiber mat-matrix interface, corroborating its superior mechanical performance. Thermal analysis (TGA) indicated that the S9 nano silica biocomposite exhibited the least weight loss (79.4 wt%), demonstrating superior thermal strength and resistance to temperature degradation compared to other samples (S1-S13). These results suggest that bamboo fiber mats and nano-silica biocomposites are particularly suitable for manufacturing packaging cases and domestic lightweight applications. Their improved mechanical strength and heat resistance make them well-suited for these applications, offering an eco-friendly alternative to conventional materials. Future research should focus on optimizing processing parameters and exploring additional applications for these innovative biocomposites, furthering their potential in sustainable material development.

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