Mechanical and Dynamic Mechanical Characterization of Epoxy Composites Reinforced with Casuarina Leaf Bio Fibre

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Abstract: This study investigates the influence of casuarina leaf biofibre on the mechanical and morphological characteristics of epoxy resin composites. Composites were prepared using the hand-layup method, incorporating varying volume fractions of the biofibre (4%, 8%, 12%, 16%, 20% and 24% v/v). Mechanical and dynamic mechanical tests were carried out to assess the impact of the biofibre on the composite material. Mechanical testing revealed significant enhancements in tensile strength of 29.2 MPa, particularly at a biofibre volume fraction of 12% and the composite with the highest impact and flexural strengths measured at 2621 J/m^2 and 41.1 MPa, respectively for the 16%v/v fibre loading. Dynamic mechanical analysis (DMA) results demonstrated improved viscoelastic properties attributed to the presence of the biofibre. Scanning electron microscopy (SEM) examination of tensile tested samples provided insights into interfacial bonding and filler dispersion within the composite matrix. Overall, the findings highlight the potential of casuarina leaf biofibre to enhance specific mechanical properties and viscoelastic behaviour of epoxy casuarina composites. Optimizing the biofibre volume fraction offers possibility to tailor composite properties for specific application requirements. This research advances the development of bio-based composite materials, contributing valuable insights for the creation of sustainable and high-performance engineering materials.

Keywords: Epoxy resin, composite, casuarina leaf fibre, morphological analysis.

1. INTRODUCTION

The use of biofibres as fillers is increasingly popular due to low cost, flexibility, eco friendly and specific stiffness, with biofibres-reinforced composites gaining acceptance in structural applications, household application. As technological advancements and consumer demands drive the need for sustainable materials, biofibre composites have evolved significantly, becoming versatile engineering materials with a broad range of properties, though they face continuous competitive pressure and require ongoing research [1]. Researchers have investigated the physical, mechanical and thermal behaviour of the glass filler hybrid matrix composites, highlighting the importance of filler geometry in enhancing the mechanical properties of composites [2]. In the work by Kavimani *et al.* [3], the addition of bio-exfoliated graphene oxide (BGO) to the polymer resulted in significant enhancements in the material's mechanical properties such as tensile, fleuxural and impact strengths, with increases of up to 55%, 28%, and 80% respectively. The mechanical properties of the polymer composite were enhanced by precisely regulating the amount of BGO particulate filler. The referenced study delves deeply into the precise proportion of biofiller required for optimal qualities. Matykiewicz [4] investigated the effects of adding biochar at weight percentages of 2.5%, 5%, and 10% to particulate carbon fibre incorporated epoxy composites. With an addition in the biochar content, the flexural strength rose from 275 MPa to 323 MPa, suggesting that filler with about 10% biochar could have superior mechanical properties. According to Mahesha *et al.* [5], a composite containing 30% hemp, 7% jute, 57% epoxy, and 6% TiO_2 showed significant improvements in mechanical properties. The composite's tensile, flexural, and impact strengths improved by 24.21%, 25.03%, and 24.56%, respectively. These findings point to the optimal epoxy composite composition for improved mechanical properties. Although the precise

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percentage for ideal properties was not specified, Irawan [6] demonstrated that adding Rudraksha seed filler improved the mechanical properties of polymer composite materials. More research is needed to determine the precise filler percentage that produces the best mechanical properties. Toorchi *et al*. [7] reported that a composite containing five weight percent NaOH-treated fibre and five weight percentage nanoclay exhibited significant enhancements in mechanical strength. However, the appropriate proportion of bio filler was not stated.

Loeffen *et al*. [8] reported significant improvements in mechanical properties with 0.3 weight percent graphene oxide (GO) filler in bio-epoxy composites, including a 23% rise in tensile strength, a 35% rise in tensile modulus, a 17% increase in flexural strength, and a 31% increase in flexural modulus. This percentage was found to be optimal for these characteristics. Mulenga [9] reported improvements of 6.3% in tensile strength, 68% in flexural strength, 28% in impact strength, and 17% in scratch hardness for hybrid composites with 20% fibre weight and 5% fly ash weight. It was found that the better improvements in mechanical properties came from this composition. The highest flexural strength of 150.45 MPa and tensile modulus of 8.4 GPa were shown by composites containing 40% flax fabric, suggesting the ideal filler percentages for different properties. The ideal composition for epoxy composites with hollow glass microspheres (HGM) and nano-fillers (graphene and carbon nanotubes) is 10% HGM and 0.5% weight of graphene, according to Kumar *et al*. [10]. The combination produced greater flexural and tensile moduli, but impact strength declined with increasing HGM content. Borhan [11] studied the effects of tensile testing on unsaturated polyester casuarina composites at varying loadings from 10 to 50 wt%. The results indicated that as the filler loadings of casuarina leaves increase, the tensile modulus decreases, with the highest value being 1.6 GPa at 10% w/w and the lowest value being 1.3 GPa at 50 wt%. The study investigated the influence of 1, 5, and 10 wt% natural fillers (lignin, chitosan, starch) on the thermal and mechanical properties of epoxy resin composites, found that 5 wt% chitosan caused the greatest decrease in the modulus of elasticity, while at 10 wt%, starch led to the largest decrease and chitosan slightly increased the modulus from 2158 MPa to 2318 MPa compared to its 5 wt% filling [12]. Yesuraj *et al.* [13] found that incorporating Almond Shell powder into an Aquilaria Agallocha Roxb Blended Epoxy Hybrid Matrix improved mechanical properties up to a 20% volume fraction, where maximum tensile, flexural, and impact strengths were observed, while 15% provided the best visco-elastic properties; however, further increases in filler content led to reduced mechanical performance due to non-uniform dispersion and filler accumulation. Chee *et al*. [14] utilized TMA and dynamic mechanical analysis DMA to study how hybridizing bamboo and Kenaf fibers affects the thermal properties of epoxy resin-based composites, finding that the composite with 100% bamboo achieved a storage modulus (E′) of 979 MPa, compared to 449 MPa for pure epoxy resin and 775 MPa for 100% Kenaf.

According to George and Mohanty's [15] research, adding 0.75 weight percent graphene-decorated graphene quantum dot (GDGQD) to epoxy composites significantly increased the composites' flexural strength (31%), impact strength (102%), compressive strength (22%), and compressive modulus (29%). This was the ideal filler concentration to improve mechanical properties. According to Huang Na *et al*. [16], fillers made of herbaceous plant biomass had no discernible impact on the PLA composites' flexural strength. Nayak and Mohanty [17] compared the mechanical characteristics of hybrid polymer composites, including impact strength, flexural strength, and lap shear strength; however, they did not provide precise enhancements or recommended bio filler proportions. When used as a micro-filler in epoxy composites, oil palm ash (OPA) demonstrated the best mechanical properties at 30% filler content of 300 mesh-size filler, according to Rizal *et al*. [18]. At this concentration, the maximum flexural and tensile strength, tensile modulus were measured. The incorporation of biofibre such as graphene oxide, biochar, nano-clay, natural fibers, and agricultural waste significantly enhances the mechanical properties of epoxy composites. The optimal filler percentages vary based on the type of filler and composite application. These findings underscore the potential of bio fillers in developing high-performance, sustainable composite materials. Recent advancements underscore the growing importance of studying the mechanical behavior of natural fiber reinforced composites to ensure appropriate selection according to end-use applications, as it plays a crucial role in various composite processes. Casuarina equisetifolia, commonly known as She-oak, Junglisaru, Ironwood is a fast-growing, evergreen tree that can reach heights of 10-50 meters and is extensively cultivated in sandy soil regions of Gujarat, Orissa, West Bengal and Tamil Nadu in India. The main constituents of casuarina leaf are cellulose, hemicellulose, lignin and pectin [19].

Cellulose and lignocellulose fibers are the most commonly used natural fibers due to their low cost and superior mechanical properties compared to other natural fibres [20]. In this study, casuarina biofibre was selected to investigate mechanical and dynamic mechanical properties due to their relatively low cost, renewable characteristics, availability and ability to enhance the properties of composite materials.

2. EXPERIMENTAL PART

2.1. Preparation of Epoxy Casuarina Composites

Epoxy resin (LY556) matrix material and polyamine-type (HY951) hardener were used in this study. The biofibre used in this work was casuarina leaf fibre obtained from Vimal Agencies, Kanchipuram, India was dried and ground with a grain size of 10 to 25 microns using ball milling process. The density of casuarina bio fibre powder was found to be 1.05 g/cm³. Hand layup technique followed by compression was used to produce composite samples. The samples were fabricated by changing the volume percentage of casuarina leaf fibre as 4%, 8%, 12%, 16%, 20%, and 24% v/v which is incorporated into the epoxy matrix to study the influence of the biofibre content on the mechanical and biodegradable properties of the composite material. The hand layup method was used to fabricate the composite samples. The interior of the mould was coated with a release agent to facilitate the removal of the cured sample. The weight ratio for combining the epoxy resin and hardener was 10:1. The casuarina fibre was added to the epoxy mixture using preset volume fractions. The mixture was manually stirred for 15 min to ensure even distribution of the filler in the resin. The mixture was then poured into a wooden mould and smoothed using a brush. Finally, the cured composite was cut into samples according to the ASTM dimensions in random directions to ensure the uniformity of the test specimens.

2.2. Characterization

2.2.1. Mechanical Testing

Tensile, impact and flexural tests were performed on the epoxy composite specimens in compliance with ASTM guidelines. Tensile testing was carried out at a cross-head speed of 5 mm/min while adhering to ASTM-D638 guidelines. A three-point bending test performed in accordance with ASTM-D710 protocol at a speed of 1.5 mm/min was utilised to determine the flexural properties. Using an IZOD Impact tester, the composite specimen's impact strength was evaluated in compliance with ASTM-D256 standards. Five samples were analysed for each test, and the average result was considered for discussion.

2.2.2. Dynamic Mechanical Analysis (DMA)

Dynamic Mechanical Analysis was utilised to examine the viscoelastic properties of the epoxy composite samples. DMA testing provides valuable insight into the behaviour of the material at various temperatures and mechanical loads. Following ASTM guidelines, the tests were performed with specific parameters chosen to match the experimental setup. The damping characteristics, loss modulus and storage modulus of the composites are all determined by DMA testing, which also sheds light on the materials' mechanical behaviour under dynamic conditions.

2.2.3. Fractographic Analysis

SEM images were captured in order to examine the morphological characteristics of the specimens. The interaction between the bio-filling material and matrix was assessed by looking at the specimens' tensile fractured surface. Hitachi's S 3400N scanning electron microscope was utilised to capture the SEM images. Prior to analysis, the samples were sliced and coated with carbon. An accelerating voltage of 20 kV was applied during the test.

3. RESULTS AND DISCUSSIONS

3.1. Mechanical Properties

Figures **1** through **3** display the outcomes of the tensile, flexural and impact tests. The graphs demonstrate a measureable influence of the volume percentage of the biofibre on the mechanical properties of the epoxy composites. The impact of the casuarina leaf biofibre on the tensile strength of the epoxy composite is demonstrated using Figure **1**. Fillers are typically added to enhance the stiffness (modulus) and toughness of composites. When a load is applied, the matrix and fillers separate, requiring energy that depends on the bonding strength between the filler and the matrix material. As shown in the figure, adding casuarina fibre up to 8 vol% results in only a slight improvement in the impact strength of the composite material. However, increasing the fibre content beyond 8 vol% leads to a moderate increase in impact strength. Further addition of filler beyond 16 vol% reduces the impact strength, though it remains higher than that of the neat resin. This reduction is attributed to the agglomeration of bio fibre in the epoxy

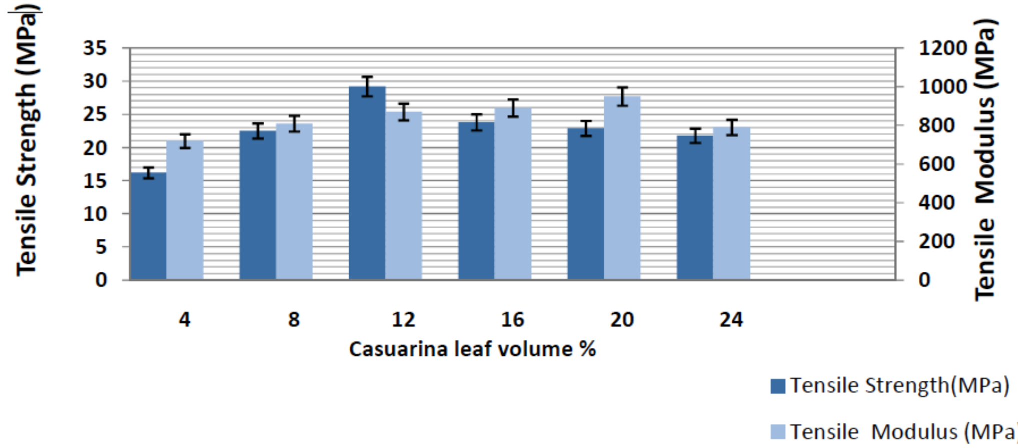

Figure 1: Tensile characteristics of casuarina leaf filled epoxy composites.

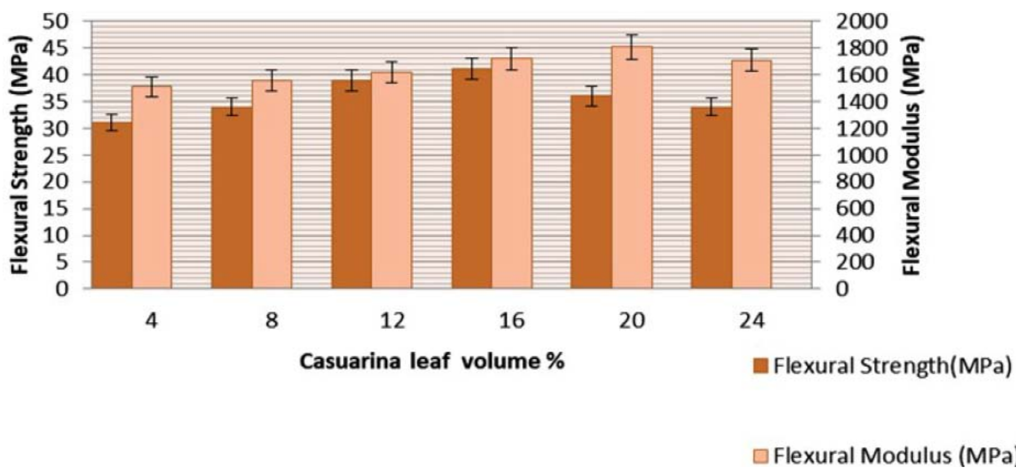

Figure 2: Flexural characteristics casuarina leaf filled epoxy composites.

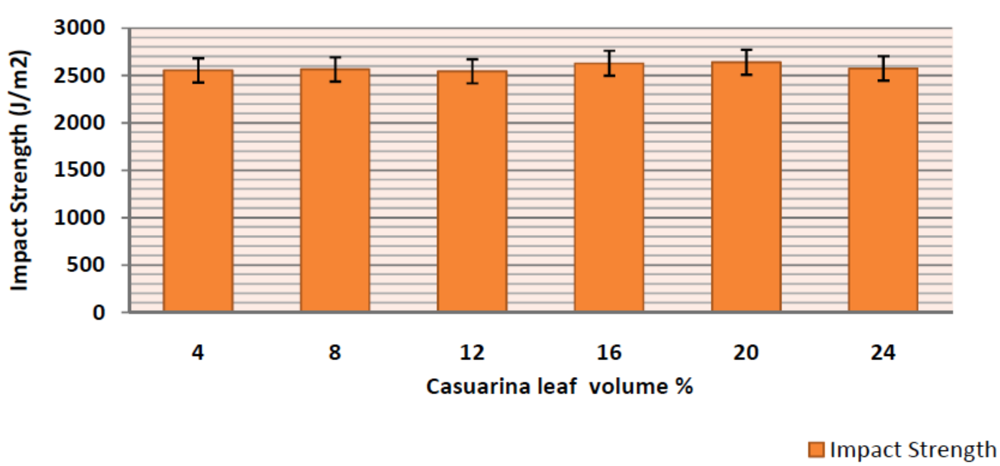

Figure 3: Impact strength of the epoxy casuarina leaf composites.

matrix, as observed in the SEM image, which results in poor tensile properties. Biofibre loading of 12% v/v brought in a peak tensile strength of 29.2 MPa for the composite. Similarly, the composite with the highest impact and flexural strengths measured at 2621 J/m^2 and 41.1 MPa, respectively for the 16%v/v filler loading. This is a result of the granular filler material, which may not significantly affect the mechanical strength of the epoxy composite as a whole. However, the tensile and flexural moduli of the composite material have improved as a result of the addition of biofibre. This indicates that by increasing the composite's stiffness and rigidity, the filler material raises the composite's tensile and flexural moduli. Notably, composites with a biofibre volume fraction higher than 20% v/v show a significant deterioration in mechanical properties. This phenomenon is caused by the absence of interaction between the biofibre composed of casuarina leaf fibre and the epoxy matrix material. The matrix bond of the filler material may be compromised, which would impair its mechanical performance. These outcomes are in line with those of Boronat *et al*. [21], who used eggshell and green polyethylene biopolymer to create a biocomposite. Their work highlights the crucial role fibre materials play in increasing the thermal, mechanical and rheological properties of composites.

3.2. Fractography Study

The SEM images displayed in Figure **4a-4h** provide important details about the dispersion and interfacial adhesion of the casuarina leaf fibre within the epoxy matrix. These micrographs show the visual interaction between the matrix and fibre. They were removed from the specimen's tensile fractured surface. The composite in Figures **4a** and **4b** contains 4% v/v biofibre. The sparse distribution of voids in the matrix indicates insufficient filler material distribution and insufficient stress transmission between the casuarina leaf fibre and the matrix. Furthermore, it is clear that in this case the matrix is stressed, indicating a weak filler-matrix interaction. Conversely, Figure **4c** and **4d** display SEM images of a composite containing 12% v/v biofibre. The images exhibit rough fracture surfaces and spaces within the epoxy matrix, suggesting that the filler material and epoxy matrix have improved adherence. This enhanced interaction implies better stress transfer across the interface, which enhances the mechanical properties. However, as you can see in Figures **4g** and **4h**, you get a different phenomenon when you go over a 20% v/v bio fibre volume. The filler surface was not adequately wet by the epoxy matrix, as the SEM images demonstrate, which resulted in poor interfacial adhesion between the two materials. Insufficient load transfer from the filler to the matrix reduces the mechanical strength.

3.2.1. EDX Analysis

EDX is a powerful analytical method for elemental analysis or chemical characterization of a sample. Materials' elemental composition is determined by the interaction of X-rays with matter. An elemental analysis technique that is popular and versatile, EDX analysis combines high spatial resolution with quick, non-destructive testing. It can be applied to a wide range of fields. Although it has certain drawbacks, its capacity to offer comprehensive compositional data makes it a priceless resource for academia and business. The elemental analysis (EDX) images are shown in Figure **5**, show that the sample is rich in carbon (81.7 wt %) and has a significant oxygen content (18.09 wt %), which indicates the presence of an organic or oxidised material. This information is needed for applications in environmental research, quality control, and material science. The high percentage of carbon might point to the presence of specific materials or processes, like organic compounds or oxidation states, when combined with the oxygen content. Further details regarding the preparation and source of the sample would enhance understanding and increase the applicability of these results.

3.3. Dynamic Mechanical Analysis

3.3.1. Storage Modulus (E')

Figure **6** shows how the storage modulus of a composite made of epoxy resin and casuarina leaf fibre varies with temperature and frequency. The experiments were carried out between 30 and 240 °C at a frequency of 5 Hz. The findings suggest that, regardless of frequency, the composite shows higher energy dissipation up to 140 °C than its constituent parts. The test temperature increases the amount of energy absorbed by the resin and the composite, indicating that filler and filaments are actively moving within the matrix up to 140 °C. Nevertheless, the composite's ability to perform as intended decreases above this temperature, most likely as a result of the polymer matrix's increased relaxation. The storage modulus or stiffness of a composite is known to improve with an increase in particulate filler content in the polymer matrix. Furthermore, treated fibre that has been filled with filler expands, improving quality and increasing the expected storage modulus over a shorter period of time. On the other hand, lower frequencies and slower relaxation lead to less stiffness because lower frequency particles rearrange more frequently than higher frequency particles. Within a certain temperature range, the material's atomic rearrangements result in a decrease in stiffness.

3.3.2. Loss Factor

The amount of molecular movement within the polymer cross linked chain and energy dissipation throughout loading are indicated by the loss factor, which is typically denoted by the product of the storage

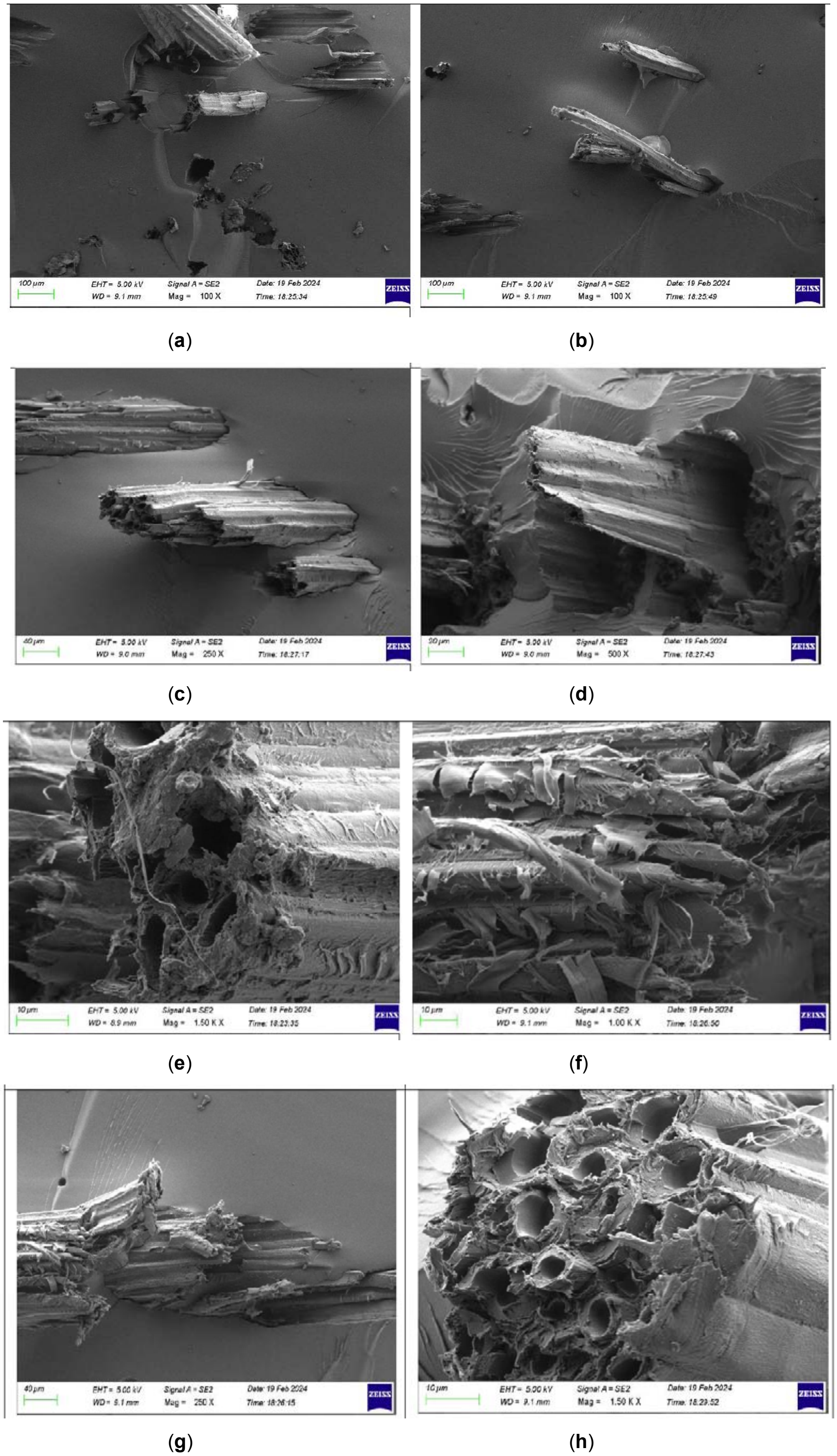

Figure 4: SEM images. (**a**) & (**b**) Epoxy composite with 4% v/v biofibre. (**c**) & (**d**) Epoxy composite with 12% v/v biofibre. (**e**) & (**f**) Epoxy composite with 16% v/v biofibre (**g**) & (**h**) Epoxy composite with 20% v/v biofibre.

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C K	81.91	85.78	9.55	844.65	0.9413	0.2149	1.0000
O K	18.09	14.22	12.97	127.74	0.9486	0.0614	1.0000

Figure 5: EDX image of the biofibre incorporated composite.

modulus and the loss modulus. The loss factor plot for the composite material made of casuarina leaf fibre is given in Figure **7**. The findings infer that additional incorporation of reinforcement lowers the composite material's loss factor, suggesting that the material prefers energy dissipation over stiffness. The substance is thought to be in its solid state at low temperatures, when it dissipates energy effectively. The material goes through phase transitions to an elastic or viscoelastic state as temperature rises. The glass transition temperature (Tg), also known as the temperature at which the maximum loss modulus (E″ max) is observed, is the temperature at which these transitions take place. The glass transition temperature in this study is determined using the maximum tan value. The influence of filler in the polymer composite results in a discernible difference in the glass transition temperature (Tg) when related to epoxy resin. This illustrates how the interaction between matrix and fibre affects Tg.

3.4. Biodegradability Study

The epoxy composite containing biofibre was evaluated for biodegradability using the ASTM D5988 standard test protocol. This method involves putting the specimens through carefully controlled environments that closely mimic natural conditions in order to assess how the specimens deteriorate over time. The specimens were exposed to environmental factors that would facilitate biodegradation, such as favorable temperature and humidity, and their weight changes were routinely monitored. The weight loss that has been seen over time provides valuable information about the sustainability, environmental impact, and degree of biodegradation of the composite. The test results given in Figure **8** show that the specimens' weight increased by a small amount during the first week due to moisture absorption. However, there is a noticeable weight loss in later stages, which indicates that the biofibre material is degrading. This

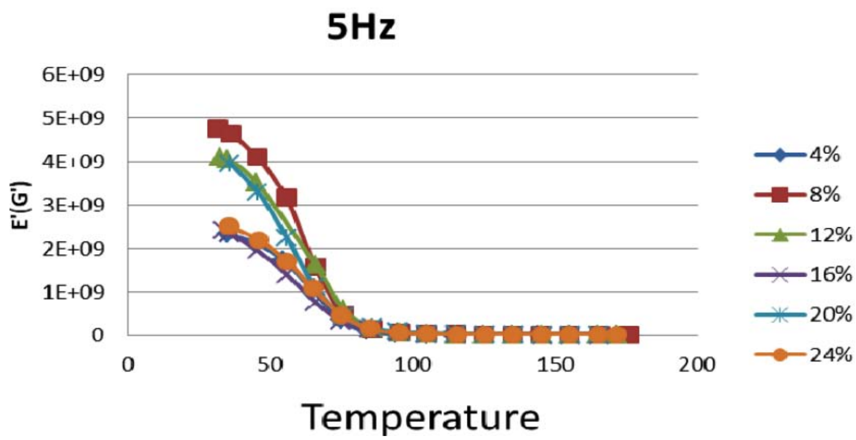

Figure 6: Storage modulus of epoxy casuarina leaf fibre composite at 5 Hz.

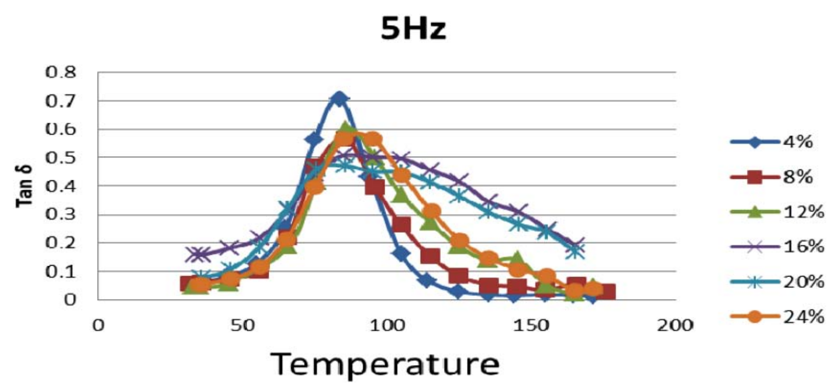

Figure 7: Tan δ of epoxy casuarina leaf fibre composite at 5 Hz.

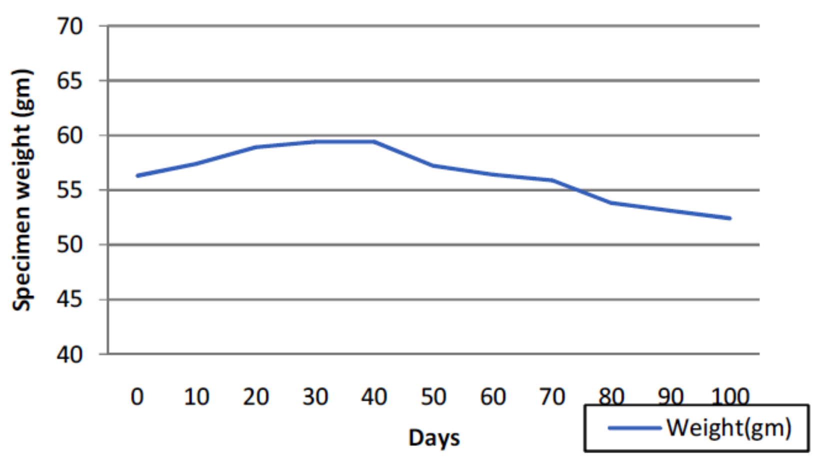

Figure 8: Plot of specimen weight with respect to time in biodegradability test.

phenomenon emphasizes the possibility of a natural breakdown of the epoxy composite containing biofibre.

CONCLUSIONS

The mechanical properties of epoxy casuarina leaf fibre composites are greatly enriched by the addition of casuarina leaf biofibre, as demonstrated by tensile, impact, and three-point bending tests. A 12% v/v and 16% volume fraction of bio filler is used to balance matrix compatibility and reinforcement for the best mechanical performance. On the other hand, composites with filler volume fractions higher than 20% v/v show diminishing returns because of insufficient filler-matrix interaction. These results highlight the significance of biofibre content and ensuring strong interfacial adhesion in order to maximise mechanical performance. SEM analysis further highlights the importance of fibre content in influencing epoxy composites' dispersion and interfacial adhesion. Inadequate mechanical strength and interfacial adhesion result from high filler content. These findings underscore the potential of casuarina leaf biofibre to provide better mechanical properties of epoxy based composites, providing better understandings for composite design and manufacturing processes across various applications.

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DECLARATION OF COMPETING INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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