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Mechanical, dynamic mechanical and thermal analysis of casuarina leaf fibre and moringa gum filler reinforced epoxy biocomposite

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These days, the automotive sector and manufacturing industries have begun adopting renewable materials due to public awareness of the usage of polymers and severe legal pressures surrounding their use. This research is focused on using casuarina leaf fibre as reinforcement in an epoxy matrix together with natural moringa gum filler since natural fibre reinforced composites are essential for the development of lightweight structural materials. It is observed that the synergistic impact of fibre and natural filler improved adhesion and stress is transferred more evenly across the reinforcements. The mechanical, viscoelastic, thermal and biodegradability properties have been investigated on incorporation of 4,8,12,16 and 20v/v % of moringa gum in casuarina epoxy composite and the results indicated that the addition of moringa gum filler increased the tensile, flexural and impact strength of the composites indicating that moringa gum is a promising filler. Using thermogravimetric analysis, Differential Scanning Calorimetric analysis, it is found that the development of hybrid composites increased thermal stability, with distinct degradation patterns.

Keywords: Casuarina leaf, Epoxy bio composite**,** Flexural and impact strength, Moringa gum filler, Tensile strength

Introduction

In the current era, polymer composites are gaining notable attention in various engineering fields, such as construction, automobiles, aircraft, and space, due to their superior strength-to-weight ratio when natural fibres are used as reinforcement. Ongoing research is focused on developing cost-effective, eco-friendly composite materials using widely available natural fibres, which allow for easy modification of properties in reinforced polymer composites. Bio-based fillers in polymer composites are attractive because they improve mechanical characteristics, thermal stability and sustainability. Bio fillers in composites reduce environmental degradation, carbon emissions, energy use, waste and pollution and create a circular economy. Global efforts to minimize non-renewable synthetic fibres and fillers are supported by bio fillers in composite materials. Many researchers have investigated using natural bio-derived fibres as polymer-based composite reinforcements[1–3].

Plant fibres, with their high thermal and mechanical properties offer a sustainable and cost-effective alternative to synthetic fibres, which can significantly alter the polymer's molecular and microscopic characteristics. Natural fibres like hemp, jute, and flax are increasingly valued in engineering uses, such as automotive and aerospace, for their strength, availability, corrosion resistance, nonabrasive nature, biodegradability, and superior acoustic and thermal insulation, despite challenges like moisture absorption and processing temperature[4].

Epoxy is widely used in industries like coatings, adhesives, electronics, and aerospace because it has strong mechanical and chemical properties, resists shrinking during curing, handles heat well, and works effectively as a matrix material. Natural fibres, extracted from both primary sources like sisal and jute, and secondary agro-waste sources like coir and rice straw, are widely used in various applications[5].

The enhanced strength of the composites is ascribed to the fibres' ability to endure stresses transferred from the polymer matrix, with the Cardanol/bagasse fibre composite exhibiting superior

performance due to the absence of voids, well-embedded fibres, and strong adhesion at the fibre–matrix interface[6]. Incorporating moringa gum into the epoxy matrix enhanced tensile, flexural, and impact strength, attributed to the strong interfacial interaction between the resin and the gum[7]. The epoxy composite with 20% *Moringa oleifera* leaf showed better tensile and flexural strength, along with improved thermal stability, breaking down at a peak temperature of 410°C as shown by the DTG[8]. Adding 12% powdered *Moringa oleifera* fruit pod to epoxy resin improved the bio-composite's flexural strength, modulus, tensile modulus and hardness by 28.7%, 25%, 8.8%, 67.9%, respectively[9]. Incorporation of casuarina leaf fibre into epoxy matrix increased the tensile, flexural and impact strength due to the good interaction between the resin and the fibre[10]. Epoxy-based composites containing teak wood dust (450 μm) at varying weight fractions (10%, 15%, and 20%) along with 5% chopped chicken feathers, fabricated using the hand lay-up method, exhibited the highest strength of 8.93 MPa at 15% teak wood dust, while strength decreasing beyond this proportion[11].

Epoxy composites supplemented with hemp fibres increased tensile strength and flexural modulus by 15%, according to Gargol *et al*.[12]. As a stiff and strong natural fibre, hemp improved fibre-matrix adhesion and reduced composite brittleness. Due to superior load distribution between the matrix and fibres under stress, hemp-reinforced composites have higher elasticity and strength. These findings implied that hemp fibres offered significant mechanical and environmental benefits in composite design. Deepak *et al*. examined the dry floral waste bio filler effect on epoxy composite mechanical characteristics[13]. This study examined bio fillers' impact strength, flexural properties, and tensile properties with varying volume fractions. Tensile (24.9 MPa) and flexural (39.1 MPa) moduli improved with bio filler, especially at 12% v/v. Khademi *et al*. [14] predicted a 30% increase in tensile strength for natural fibre reinforced epoxy composites using a strain-rate dependent micromechanical model. The model showed that greater strain-rates increased load-bearing capability by quantifying load transmission between fibres and matrix. At ideal strain-rates, flax and hemp showed higher mechanical performance, demonstrating the importance of processing conditions on composite mechanical qualities. Pradhan *et al*.[15] studied using bio waste pistachio nut shell particles as a strengthening medium in epoxy-based polymer composites. In their study investigating tribological behaviour and filler content on abrasion resistance, the composite with 20 wt% filler performed best. Pistachio shell particles greatly boosted the composites' abrasion resistance. Wood apple shells and pine cones were tested as bio-based epoxy composite fillers by Joshi *et al*.[6]. This study evaluated how filler loadings affected density, void fraction, mechanical strength, morphology, and moisture absorption. According to reports, bio filler structure and content affected composite mechanical properties. Pine cone fillers increased flexural strength and modulus, while wood apple shell fillers increased tensile, impact, and interlaminar shear strength the most. Sukania *et al*.[17] found 62.3 MPa and 8.72 GPa tensile strengths and moduli of elasticity in banana fibre reinforced epoxy composites. Osorio *et al*.[18] found 33 MPa transverse three-point bending strength for untreated bamboo fibres in epoxy composites. Alkali treatment of bamboo fibres enhanced mechanical characteristics by removing surface contaminants and increasing fibre roughness, resulting in better micromechanical connection between fibres and matrix. Bamboo fibres, with their high strength-to-weight ratio, enhanced the flexural capabilities of composites, making them excellent for lightweight, robust applications. Belhadj *et al*.[19] examined alfa fibre-reinforced epoxy-amine composite mechanical characteristics. Fibre content improved mechanical qualities, however excessive fibre content decreased performance due to poor dispersion and fibre-matrix adhesion. This study emphasized the necessity of optimising fibre content for mechanical qualities and showed how fibre treatment affected natural fibre composite viscoelasticity.

Incorporating *Washingtonia filifera* (WF) fibre reinforcement into a high-density polyethylene (HDPE) matrix enhanced the thermal stability of the HDPE-WF bio composites, particularly maximum effectiveness achieved for 20% WF by mass, with main degradation occurring between 391 and 492°C. The thermal stability of these bio composites surpassed that of lignocellulose materials, which degraded between 150 and 500°C, and the highest residue was observed in the HDPE-30WF and HDPE-20WF bio composites, showing 7.01% and 5.17% residue at 600°C due to char formation from hemicellulose and lignin degradation[20]. Increasing the kenaf fibre content in the

epoxy polymer improved the thermal stability of the composites, as shown by the higher char residue at 900°C, particularly for those with 20 vol% and 30 vol% kenaf fibre. The addition of kenaf fibre delayed the total degradation of the composite, resulting in greater residue at 900°C compared to the char residues of kenaf fibre and epoxy. Factors like chemical composition, crystallization, oxidation, filler content can change the glass transition and behaviour of polymers, and in kenaf fibre epoxy composites, the introduction of fibres primarily restricted the macromolecular mobility of the epoxy, impacting its thermal behaviour [21]. Incorporation of DPF (date palm fibres) into the epoxy enhanced the heat stability of 50% DPF/epoxy composites, with the highest residual content (19.8%), which enhanced thermal stability and flame resistance compared to 40% DPF and 60% DPF loadings[22].

The inclusion of 6% coconut fibre with 20% almond shell particle-reinforced epoxy bio composite enhanced its thermal stability compared to bio composites made solely from almond shell particles[23]. Souza *et al*.[24] investigated how varying the percentage of caranan fibre from 0% to 30% affected the thermal characteristics of natural composites. Their findings revealed that increasing the caranan fibre content raised the glass transition temperature (T_g) of the composites, with 20% and 30% fibre contents resulting in Tg values of 96°C and 113°C, respectively. The TGA results studied by Boopalan [25] revealed that the thermal stability of epoxy composites was influenced by the fibre type and ratio, with significant weight loss occurring between 376 and 380°C due to epoxy and fibre degradation. Basha *et al*. [26] explored the properties of epoxy resin reinforced with sugarcane fibre and millet (ragi) filler. The hybrid composite, made from 50 g of epoxy, 4 g of ragi, and 2 g of sugarcane fibre, showed excellent tensile, flexural, and impact strength, as well as a decomposition temperature above 270°C and a desirable glass transition temperature.

Model-fitting, a well-recognized method in solid-state kinetics, involves assuming a reaction mechanism, applying it to TGA data, and selecting the most appropriate model based on the quality of the regression fit. Mahmood *et al.* [27] examined the thermal degradation of lignocellulose materials such as rice husk, bagasse and wheat straw using the Coats–Redfern model, which identified D1 (one-dimensional diffusion) as the best fitting model, indicated by a high R^2 value. The activation energy increased in the order of rice husk > bagasse > wheat straw, with bagasse exhibiting the highest Kinetic activation energy (E_a) value, possibly due to its higher volatile content and lower ash content compared to rice husk. The E_a value is an important parameter for assessing the heat stability of natural fibres, typically ranges between 60–170 kJ/mol, with *Furcraea foetida* leaves having E_a of 65.64 kJ/mol, which was lower than *Prosopis juliflora* (76.72 kJ/mol) and *Cissus quadrangularis* root (74.18 kJ/mol), and similar to *Lygeum spartum* (68.77 kJ/mol)[28].

With the rise of cutting-edge industries, the need for advanced materials has led to the development of hybrid composites, which combine different fillers to achieve superior properties unattainable by using a single material. Hybridization has proven highly effective in enhancing composite properties, making the use of varied reinforcement materials increasingly common, especially in applications involving a wide range of temperature variations. Moringa gum, a natural exudate from the *Moringa oleifera* (drumstick or Miracle Tree) of the Moringaceae family, was used as a bio-filler. It is a white, sticky substance that darkens upon exposure to air, swells in water, and consists mainly of carbohydrates (62%) and proteins (7%)[29]. The gum, which is non-toxic, biodegradable, and eco-friendly, was air-dried, powdered, and stored for use.

Casuarina leaf bio fibre obtained from *Casuarina equisetifolia* contains cellulose, hemicellulose, lignin and pectin. The use of bio fibre in various fields reduced material cost, fabrication cost and repair cost. Casuarina a renewable reinforcement in epoxy composite is used due to its flexibility and availability. The fibres, with a density of 0.5–0.6 g/cm³ and size ranging from 1–3 mm in length and 20–30 μm in width, contain 8.24±0.16% moisture content, 6.61±0.67% crude protein, 21.85±0.2 % crude fibre, 4.42±0.20% crude fat, 2.25±0.28% ash, 56.64±0.35% carbohydrate and 0.34±0.001% ascorbic acid[30]. These properties contribute to improved tensile strength, stiffness, impact resistance, and overall performance of epoxy composites while reducing costs. Earlier research predominantly focused on single-type natural fibre-reinforced polymer composites. However, fibre hybridization, which combines two or more different fibres to harness their individual benefits and achieve a synergistic effect, is now gaining significant attention among researchers.

Experimental Section

Preparation of Epoxy Hybrid Composite (EHC)

To prepare the hybrid bio composites, epoxy resin [LY556] and hardner [HY-951] were purchased from M/s. Javanthee Traders, Chennai. The epoxy characterized by a density of 1.15 to 1.20 g/cm³ was utilized with the HY951 hardner which exhibits a density of 0.97 to 0.99 g/cm³ to produce composite. The hand lay-up method is the most straightforward and cost-effective method for producing relatively simple shapes. It involved using a simple open mold to shape and size the composite plates, with plastic sheets and a release gel (usually silica gel) applied to facilitate easy removal. At first the milling process of moringa gum was conducted for duration of two hours, resulting in a powder with a particle size ranging from five to fifty microns and fibres derived from casuarina leaf were systematically sectioned into a fibre length of 15 mm. Layers of resin and reinforcement (casuarina fibre + moringa gum filler) were placed in the mold. Uniform wetting and air bubble removal were achieved by pressing the fibre with a roller, and the mold was then tightly closed. A 50 kg weight was placed on the mold to apply compression, and it was left undisturbed at room temperature for 24 h to cure. After curing, the composite sheet was removed from the mold; this method's primary advantage is its low initial capital requirement. The incorporation of moringa gum was conducted at volume fractions of 4%, 8%, 12%, 16% and 20%.

Mechanical and thermal study

Tensile, flexural, and impact tests were performed, with five specimens evaluated for each filler content to ensure the statistical reliability of the results. Dynamic Mechanical Analysis (DMA) over a temperature range of 30–180 °C at 10Hz frequency and Thermogravimetric Analysis (TGA) from 30 to 600 °C under nitrogen atmosphere were employed to study the thermal properties. Morphological characterization was performed using a Zeiss Scanning Electron Microscope (SEM) to observe filler dispersion in the polymer matrix.

Kinetic study

The activation energy was calculated using the models given in Table 1. The Coats and Redfern method is considered the most versatile for calculating kinetic parameters of the degradation process and a selected degradation function f(x), the apparent activation energy E_a and frequency factor A can be determined, with various models chosen for f(x) to analyze the degradation reaction type as listed in Table 2.

Regression analysis for each model in Table 2 was conducted using Origin Pro software on all the produced composites. The model with an R^2 value closest to 1 was considered the most suitable, focusing on the main decomposition stage, which corresponded to the highest mass loss, to assess the composites' thermal characteristics. Eqs (1)-(3) were used to determine the enthalpy, entropy and Gibbs free energy of the composite.

$$\Delta S = R \ln (Ah/K_B T) \quad \ldots (1)$$

$$\Delta H = Ea - RT \quad \ldots (2)$$

$$\Delta G = \Delta H - T\Delta S \quad \ldots (3)$$

K_B (Boltzmann constant) = 1.3806×10^{-16} erg/deg/mol and R = 8.314 J/K/mol

Table 1 — Coats, Broido and Horowitz Equations

Approximation	Equation	Source
Coats- Redfern	$ln\frac{g(\propto)}{T^2} = ln\frac{AR}{\beta Ea} - \frac{Ea}{RT}$	[31]
	X axis 1/T and Y axis = $ln\frac{g(\propto)}{T^2}$	
Broido	$\ln[\ln(\frac{1}{y})] = -(\frac{Ea}{RT}) + \ln\frac{RATm^2}{Ea\beta}$	[32]
	where, , $y = \frac{w_t - w_f}{w_o - w_f}$	
	X axis= 1/T and Y axis= $\ln[\ln(\frac{1}{y})]$	
Horowitz-Metzger	$\ln(-\ln(1-\propto)) = \frac{Ea\theta}{RTs^2}$	[33]
	θ =T-Ts	
	X axis= θ and Y axis = ln (−ln 1−□)	

Biodegradability test

Biodegradation was assessed using the ASTM D5988 methodology, with specimens prepared according to standard dimensions and their initial dry weights recorded. They were buried in controlled soil conditions to simulate natural decomposition, then periodically retrieved, cleaned, dried, and weighed to calculate the percentage of biodegradation over time.

$$\text{Bio degradation (\%)} = \frac{W_i - W_f}{W_i} \quad \ldots (4)$$

Where, W_i - Initial weight of the test sample (g), W_f - Final weight of the test sample after burial (g)

Table 2 — Mathematical expressions for various models[34]

Models	Code	g(α)
One Dimensional Diffusion(ODD)	D1	α^2
Diffusion control Model (Jander)	D3	$[1-(1-\alpha)\ 1/3]^2$
Diffusion control Model (Crank)	D4	$1-(2/3)\alpha-(1-\alpha)^{2/3}$
1st order Model (Reaction order model)	F1	$-\ln(1-\alpha)$
2nd order Model (Reaction order model)	F2	$(1-\alpha)^{-1}-1$
Contracting cylinder Model(Geometrical model)	R2	$1-(1-\alpha)^{1/2}$
Contracting sphere Model (geometrical model)	R3	$1-(1-\alpha)^{1/3}$
Mampel Power Law (n=1/3)	$MPL^{1/3}$	$3[1-(1-\alpha)^{1/3}]$
Mampel Power Law (n=1/2)	$MPL^{1/2}$	$2\ [1-(1-\alpha)^{1/2}]$
Mampel Power Law (n=2/3)	$MPL^{2/3}$	$3/2\ [1-(1-\alpha)^{2/3}]$
Avrami-Erofeev Equation (n=2) (Nucleation model)	A2	$[-\ln(1-\alpha)]^{1/2}$
Avrami-Erofeev Equation (n=3) (Nucleation model)	A3	$[-\ln(1-\alpha)]^{1/3}$
Avrami-Erofeev Equation (n=4) (Nucleation model)	A4	$[-\ln(1-\alpha)]^{1/4}$

Results and Discussion

IR spectrum of moringa gum and casuarina reinforced epoxy resin composite

The IR spectrum of the moringa gum and casuarina reinforced epoxy resin composite displayed bands associated with epoxy resin, moringa gum and casuarina fibre as shown in Fig. 1**.** A broad intense peak at 3365 cm^{-1} for the hydroxy stretching vibration of free and H-bonded OH groups in polysaccharides from casuarina leaf fibre was seen. Casuarina leaf fibres contain cellulose, lignin, and hemicellulose, with abundant –OH groups that improve fibre–resin adhesion through enhanced interactions. The reinforcement of moringa gum and casuarina epoxy composites was based on achieving appropriate fibre wettability through the tailored use of epoxy polymers. This improved the interaction between fillers leading to interpenetrating polymer systems stronger interfacial bonding and increased crosslinking as evidenced by the absence of the 914 cm^{-1} peak. The bands at 2929 cm^{-1} is attributed to - CH_2 vibrations related to molecules present in cellulose and hemicellulose. Peak with wavenumber at 1507 cm^{-1} represents the stretching of C=C in epoxy ring. Moreover, the peak at 1606 cm^{-1} specifies the aromatic C=C stretching vibration in lignin. The peak spotted at 1032cm^{-1} correspond to the characteristic peak of cellulose indicates the C=O stretching of glycosidic linkage. Peak at 1234 cm^{-1} represents the stretching of C-O-C in polysaccharides[35-38].

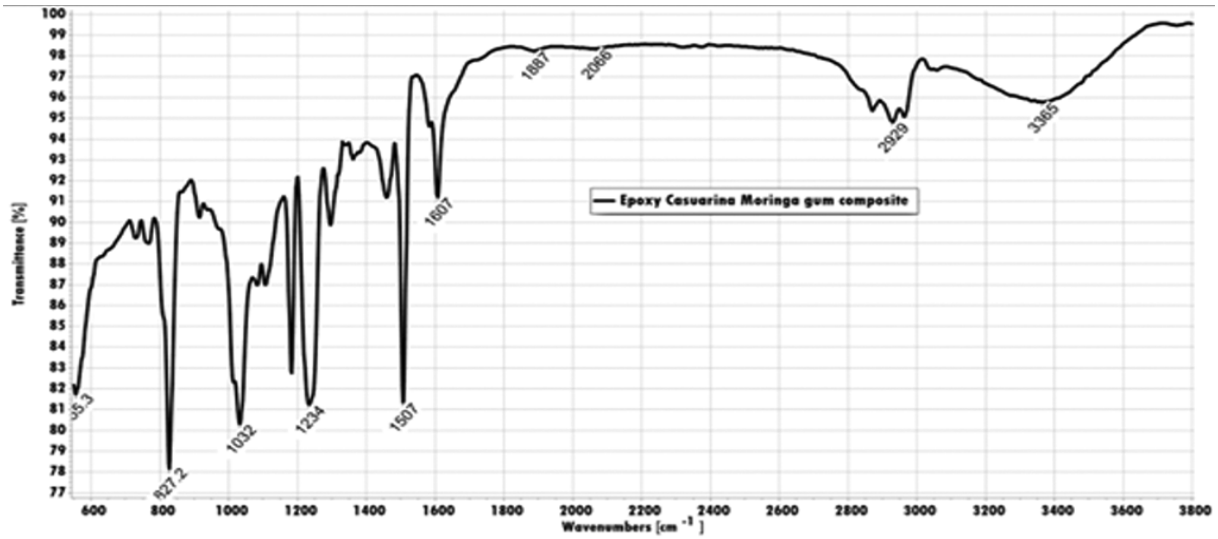

Fig. 1 — IR spectrum of Moringa gum + Casuarina epoxy composite

Elemental analysis

According to EDX analysis shown in Fig. 2, the organic composition of the composite is dominated by carbon and oxygen. The largest carbon peak (84%) at 2.88K intensity indicates that the majority of the composite is made up of carbon-based compounds from epoxy resin, moringa gum and casuarina fibre. At 0.32K, the oxygen peak (16%) is visible. Oxygen demonstrates the bonding between epoxy and bio-fillers, enhancing tensile strength, flexural strength and impact resistance. The biodegradability and low weight of the composite are guaranteed by its organic composition and absence of metallic or inorganic peaks. The carbon to oxygen (C/O) ratio of the spectrum indicates an optimal structure because oxygen functional groups enhance load transfer, decrease vacancies and promote fibre-matrix interactions. The uniform distribution of casuarina fibre and moringa gum filler in the epoxy matrix is confirmed by the absence of secondary peaks for contaminants in the EDX spectrum, ensuring consistent material quality and verifying that no unintended fillers or impurities were introduced during composite fabrication. The high carbon peak relative to oxygen for high tensile and flexural properties suggests strong matrix continuity. Oxidative cross-linking activities that enhance adhesion and heat stability are suggested by the oxygen peak at 0.32K. High carbon content and balanced oxygen integration enhance mechanical durability, chemical resilience and biodegradability in comparison to synthetic composites. High-intensity peaks demonstrate how well the processing strategy included natural reinforcements, enhancing stress distribution and impact resistance.

Mechanical properties

Figs 3-5 illustrates the tensile, flexural and impact strength of 16% v/v casuarina leaf reinforced epoxy composite with the addition of moringa gum at 4%, 8%, 12%, 16%, and 20% v/v. The tensile strength showed a significant improvement, reaching 38.5 MPa for the composite containing 16% v/v casuarina fibre and 8% v/v moringa gum. This value is considerably higher compared to the epoxy–moringa composite with 8% moringa gum alone, which recorded 12.45 MPa, and the epoxy composite with 16% casuarina fibre alone, which exhibited 29.1 MPa.[7,10]. The strong bonding between the moringa gum filler and the epoxy matrix, that promotes effective load transfer to the filler is responsible for the enhancement that was seen. The

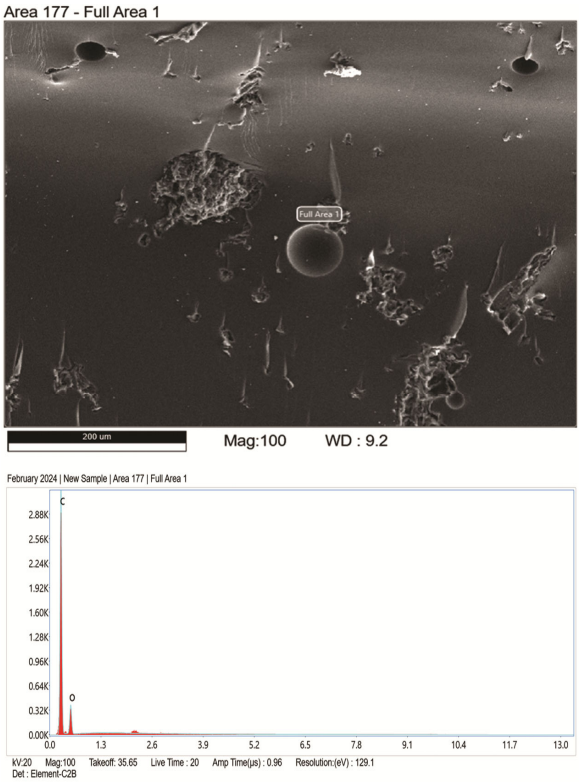

Fig. 2 — EDX mapping of the composite

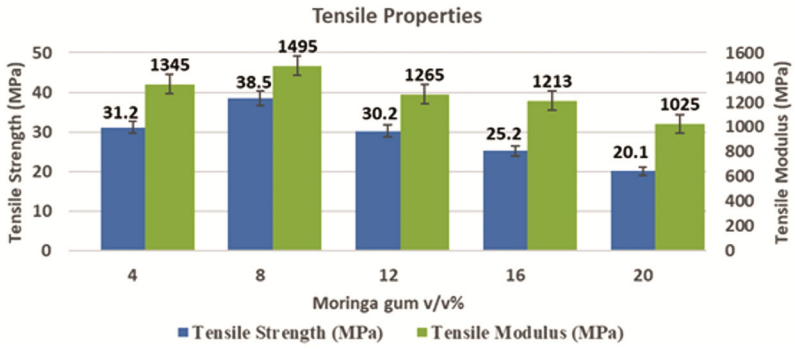

Fig. 3 — Tensile properties of Moringa gum + Casuarina epoxy composite

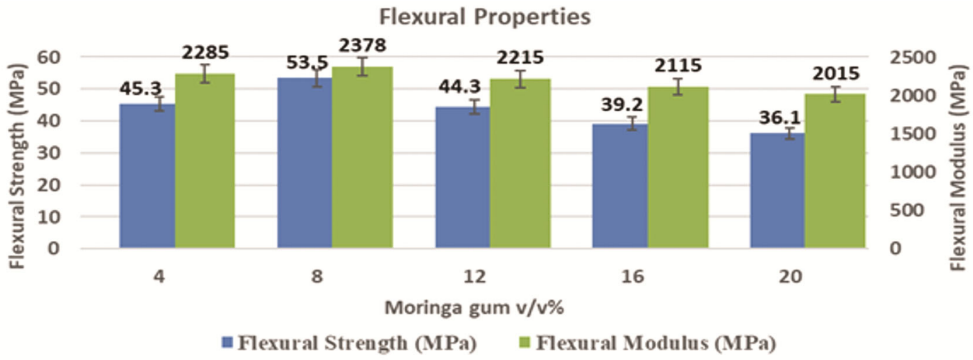

Fig. 4 — Flexural properties of Moringa gum + Casuarina epoxy composite

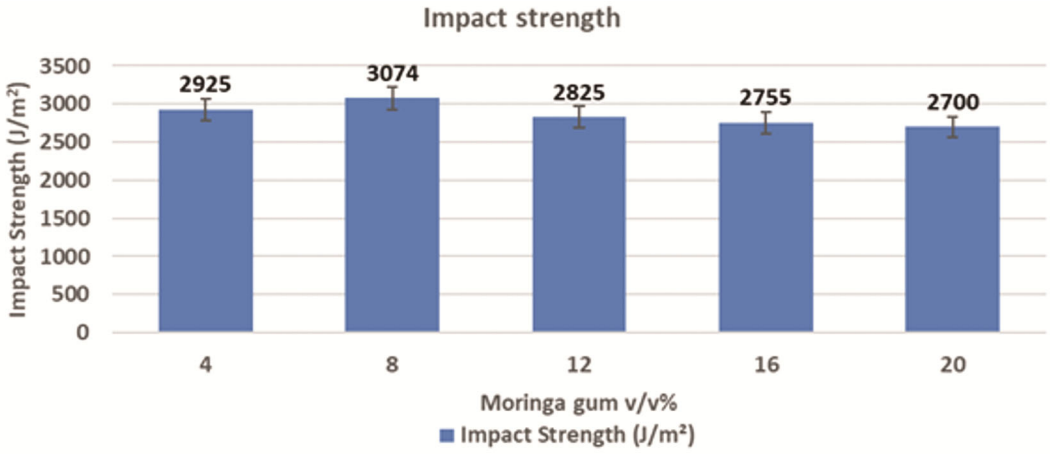

Fig. 5 — Impact properties Moringa gum + Casuarina epoxy composite

integrity of the composite material was improved and agglomeration was successfully prevented by the uniform dispersion of moringa gum particles at the determined ideal ratio.

When the concentration of moringa gum exceeded 8%, the tensile strength showed a decrease. Weak matrix interactions arise as a result of the bonding being reduced by the aggregation of moringa gum particles. This effect explains the decrease in performance when filler content rises, especially above 12 v/v% concentrations. It is noted that the overall bio filler/bio fibre content above 24 v/v % (Moringa gum and Casuarina fibre) agglomerated particles enhance the tensile failure by interfering with the uniform distribution of stress. When moringa gum is added, tensile strength is increased to an ideal loading level; but, higher concentrations can cause the filler to disperse unevenly, which could compromise the composite material's mechanical integrity[39,40].

The maximum flexural strength of 53.5 MPa was achieved for the hybrid composite. In comparison, the epoxy-moringa composite with 8% moringa gum alone recorded 18.3 MPa, while the composite with 16% casuarina fibre alone exhibited a flexural strength of 41.1 MPa[7,10]. At a loading of 8 v/v% percent, the introduction of moringa gum strengthened the stiffness of the matrix, making the composite more resilient to bending stresses. By limiting the mobility of polymer chains, the homogeneous distribution of moringa gum inside the matrix improves stiffness and deformation resistance. When the gum concentration exceeded eight percent the flexural strength showed a decrease. Filler agglomeration is a phenomenon that causes voids to occur and unequal stress distribution to be established. This ultimately undermines the framework resilience of the material, contributing a decreased performance in flexural testing values [41].

According to impact testing results the composite material showed the potential to collect energy and then release it after a quick impact event. 3074 J/m² was the highest impact strength measured for the composite with 16 v/v% casuarina leaf fibre and 8 v/v% moringa gum (Fig. 5). Epoxy moringa composite with 8% moringa gum recorded 2234 J/m² and with 16% casuarina fibre exhibited 2621 J/m² (Ref.7,10). Combining the strong reinforcing network of casuarina fibres with the crack-preventing properties of moringa gum may help achieve better energy dissipation. Because of the reduced filler-matrix interaction, lower quantities of moringa gum (4% and 8%) may result in better energy absorption and impact resistance. An increase in gum percentage of up to eight percent was shown to improve impact strength in the composite suggesting a synergistic interaction between the two that helps reinforce the matrix [42].

Impact strength was found to decrease when moringa gum was added in amounts more than sixteen percent. Particle clustering, which negatively affects stress distribution and encourages the development of localized cracks, could be the cause of this phenomenon. Since the 8% gum loading efficiently absorbs more energy, the indicated ratio exhibits beneficial features for applications needing hardness and impact resistance.

Scanning electron microscopy (SEM)

The distribution of fillers and the type of interfacial bonding inside the composites were revealed by the SEM investigation performed on broken tensile specimens. At loading levels of 8%, the SEM pictures showed a consistent distribution of moringa gum particles along with strong interfacial adhesion between the gum, casuarina leaf fibres and the epoxy matrix. When exposed to mechanical loads, the cohesive architecture improves the tensile, flexural and impact strengths of composites as well as its resistance to fracture propagation. Particle aggregation and matrix pores were visible in the SEM images of 20% moringa gum samples' given in Fig. 6. As demonstrated by the decreased mechanical characteristics at a 20% v/v, the observed flaws provide credence to the theory that higher concentrations of moringa gum impair interfacial bonding and load transfer. The synergistic effects of integrating moringa gum with casuarina leaf fibres are revealed by the mechanical characteristics of moringa gum casuarina epoxy composites.

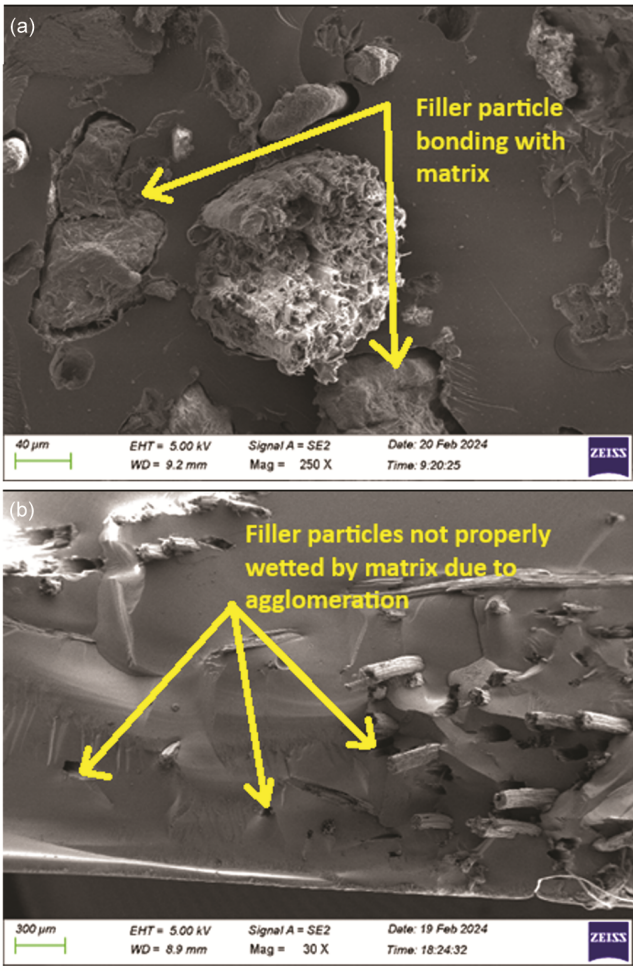

Fig. 6 — SEM images of epoxy casuarina composite incorporated with (a) 8% moringa gum and (b) 20% moringa gum

According to the investigation the better tensile, flexural and impact strengths were obtained with a gum of 8 v/v%. The addition of moringa gum initially enhances the mechanical behaviour of the composite matrix; however, agglomeration and the presence of voids ultimately reduce the overall strength, as evidenced by the SEM images.

Dynamic mechanical analysis (DMA)

DMA was employed to examine moringa gum and casuarina fibre composites' viscoelasticity at 4%, 8%, 12%, 16% and 20% v/v over 30°C to 240°C maintaining a constant frequency of 10Hz.

Storage modulus (E')

The inclusion of 16% v/v casuarina fibres significantly increased the storage modulus, attributed to the high stiffness resulting from effective stress transfer at the fibre–matrix interface, along with the fibres' high aspect ratio and superior mechanical properties that enhance the composite's load-bearing capacity. After filler (moringa gum) was included to this fibre-reinforced composite, the DMA characteristics clearly depended on the filler volume fraction and fibre-particle interactions. Effective filler dispersion throughout the matrix contributed to improve stiffness as indicated by the modest increases in the storage modulus at lower particle levels (4–8% v/v moringa gum), therefore helping to reduce localized stress transmission around fibre reinforcements. The particle filler reduces matrix chain mobility and generates small increases in viscoelastic performance by filling in gaps and increasing general matrix continuity.

The storage modulus shown in the Fig. 7 reflects the material's elastic behaviour. Storage modulus increased across all compositions, with moringa gum concentration rising to 8% v/v (E' =6.42x10^9), indicating a considerable increase in composite material stiffness and rigidity. Higher gum content (12, 16 and 20 v/v %) composites had a lower storage modulus (E') indicating faster polymer chain mobility. The composite became more flexible due to insufficient filler, which failed to form a strong matrix network. An ideal gum content of 8% moringa gum and 16% casuarina fibre produced the composite with the highest storage modulus (E') and reinforcing effect. Maximum storage modulus in 8% moringa gum, indicates powerful filler-matrix interactions for appropriate stiffness. The ideal interplay between filler and matrix explains this phenomenon where

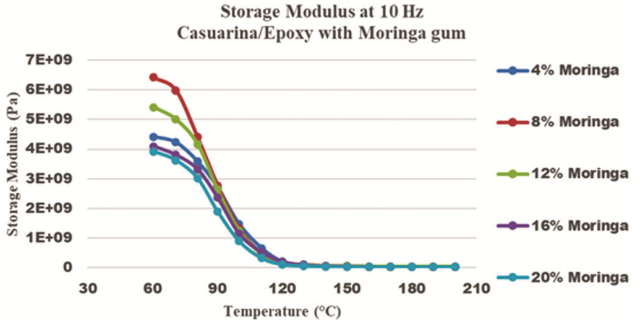

Fig. 7 — Storage modulus of Moringa gum + Casuarina epoxy composite

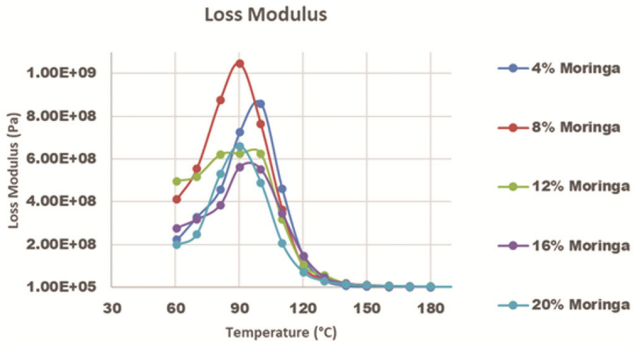

Fig. 8 — Variation of Loss modulus of casuarina/Epoxy composite incorporated with Moringa gum

fibre and gum synergistically promote stiffness and limit chain motion. Gum concentrations above 8% led to a significant reduction in storage modulus. This decrease may be due to filler agglomeration, which creates uneven stress distribution and voids in the matrix, ultimately reducing the composite's energy storage efficiency [43].

Loss modulus

The loss modulus, which measures energy dissipated as heat during deformation, determines the composite's viscous behaviour, as shown in Fig. 8. Moringa gum increased energy dissipation, as the greatest loss modulus was 8%. At 8% gum content, stress transfer and filler-matrix interaction increased the energy dissipation in the composite with the greatest E" values. The E" values at 4% moringa gum concentrations was modest, indicating little infill-matrix interaction. Insufficient infill absorbs and dissipates energy, reducing damping mechanism effectiveness. This trend matches the rise in impact resistance, as the composite absorbed more energy before failing. High gum content (more than 8 v/v%) decreased E" values. Agglomerated filler disturb energy dissipation, reducing damping efficiency.

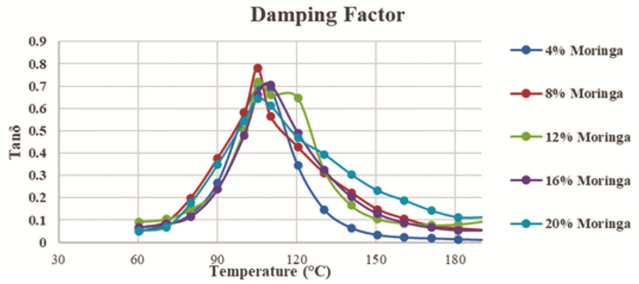

Fig. 9 — Damping factor of Epoxy Hybrid Composite

Damping factor

The damping factor (tan δ) represents the relationship between composite stiffness and energy dissipation, where a higher tan δ value indicates superior attenuation, making it suitable for vibration absorption applications.

At low gum content levels of 4% the matrix affected viscoelastic behaviour demonstrating that gum concentration was insufficient to increase attenuation (Fig. 9). The composite with 8% moringa gum showed a peak in tanδ indicating improved damping. The results show that moringa gum and casuarina fibres release energy while maintaining structure. Thermal softening occurs when the composite's storage modulus decreases with temperature, indicating a change from vitreous to rubbery. The greatest tan δ correlated with T_g, where the material transitions to a viscoelastic state under high temperatures. The thermal transition temperature of composites with a higher moringa gum concentration was significantly higher showing that the filler ingredient limited the polymer chain mobility.

Dynamic Mechanical Analysis demonstrates that the addition of moringa gum significantly improves the viscoelastic properties of casuarina fibre composites by enhancing energy dissipation, vibration absorption, and thermal stability, evidenced by a rise in T_g and reduced polymer chain mobility, making the formulation with 8% gum ideal for applications demanding high mechanical strength and damping efficiency. However, when moringa gum content exceeds the optimal level, it causes agglomeration of filler particles, which disrupts the uniform distribution within the matrix and compromises the composite's overall performance[44].

Thermal properties

The thermal study of hybrid composite on the addition of 8% moringa gum filler in 16% casuarina epoxy composite was studied as this composition showed better mechanical properties. Fig 10 shows

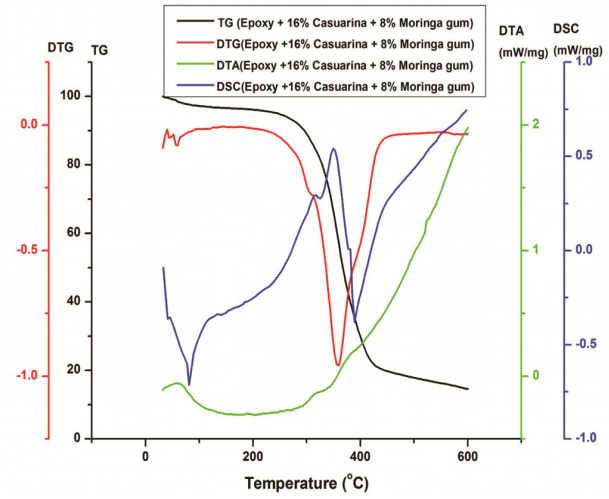

Fig. 10 — Graph of TG, DTG, DTA and DSC of 8%Moringa gum + 16% Casuarina Epoxy composite

the TGA, DTG, DSC and DTA curves of hybrid composites and monitors the thermal behaviour of the 16% casuarina leaf + 8% moringa gum epoxy composite under investigation, enabling a discussion on how the incorporation of fibre and filler reinforcement into the epoxy matrix impacts the thermal stability of the resulting hybrid bio composites. The heat resistance relies on the presence of void, filler and nature of fibre. Filler and reinforcing phases must work together to delay heat-induced deterioration to improve thermal stability. Strong interfacial bonding prevents void formation and early fibre pull-out, which accelerate heat breakdown. Char- promoting gum creates a stable protective residue. Key indicators of thermal stability include the initial, major, and final degradation temperatures, as well as the quantity of residual material formed. The initial temperature range of 30–292°C, is mainly attributed to the evaporation of weakly bound water molecules on the bio composites' surface, the dehydration of alcoholic groups, removal of volatile matter, waxy, oily substances [23,45]. The onset of thermal degradation for the hybrid epoxy composite is 293°C, which is 38°C higher than that of neat epoxy resin, while the epoxy–moringa composite with 8% moringa gum and the epoxy–casuarina composite with 16% casuarina fibre show degradation onsets at 260 °C and 286 °C, respectively[46,47]. The highest rate of weight loss occurs during the second stage, with a rapid 70.1% mass reduction between 293–496°C, primarily due to the thermal decomposition of cellulose, hemicellulose, pectin, and the glycosidic linkages in cellulose. The final step of

thermal decomposition involved the degradation of the polymer matrix occurring in the high- temperature range with a weight loss of 6.1%. High residue content of 15.5 % was formed which was far higher than epoxy. The composite's higher thermal stability resulted from the good interaction between the fibre and resin. The hybrid composite showed the highest T max (360.1 °C), surpassing that of epoxy (355.4°C) and the epoxy-casuarina composite (358°C), indicating that the addition of moringa gum enhances compatibility and structural density, thereby preserving the composite's thermal stability [48]. Thus the findings proved that the incorporation of 8% moringa gum in 16 % casuarina epoxy composite has slightly higher thermal stability than Epoxy moringa gum composite and Epoxy casuarina composite. This is due to compatibility of moringa gum and casuarina leaf fibres and sufficient interfacial bonding of fibre/ matrix in hybrid.

The hybrid composite demonstrated a superior glass transition temperature of 78.5 °C, notably higher than that of the epoxy resin (59 °C), with the epoxy+ 8% moringa composite and epoxy+16% casuarina composite recording values of 68.8 °C and 75 °C, respectively[46,47]. This could be due to the restriction offered by the fillers towards the polymeric chain mobility of the matrix during the temperature gradient.

Activation energy calculation from kinetic modeling

The model fitting method involved applying various models (Fig. 11) to the data to identify the best statistical fit from which the kinetic parameters are then calculated. The ODD model with an R^2 value of 0.9757 was selected for calculating activation energy and thermodynamic parameters and the results showed good agreement with those obtained from the Broido (Fig. 12) and Horowitz (Fig. 13) methods. The calculated activation energy (Table 3) for the addition of moringa gum to 16% casuarina epoxy composite (112.96 kJ/mol) was higher than that of 16% casuarina epoxy composite (107.54 kJ/mol). This suggests that degradation requires lot of energy. Thus, the hybridization of moringa gum filler with fibre enhances synergistic interactions among the epoxy matrix, filler, and fibre, resulting in a more stable network that demands higher activation energy for thermal degradation.

Calculation of Thermodynamic parameters

Eqs (1)- (3) were used to calculate ΔH, ΔS and ΔG and the results are summarized in Table 4. The higher

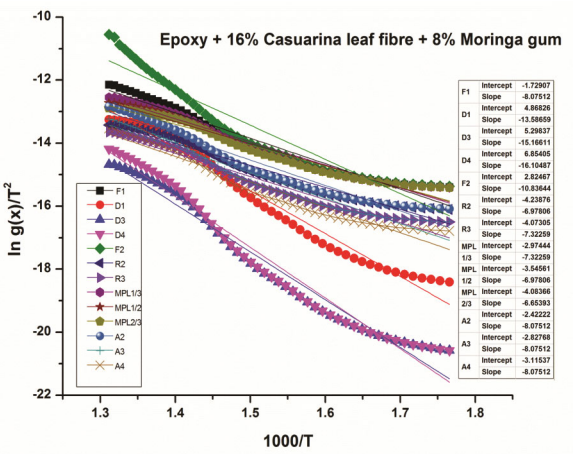

Fig. 11 — Plot of ln $g(x)/T^2$ Vs 1000/T of 8% Moringa gum + 16% Casuarina epoxy composite

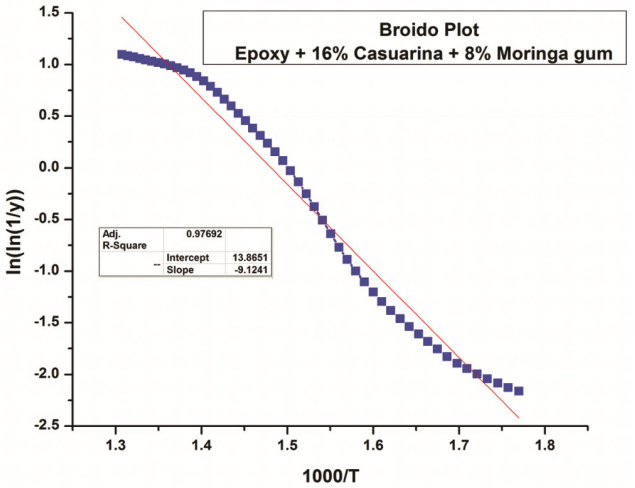

Fig. 12 — Broido plot of 8% Moringa gum + 16% Casuarina epoxy composite

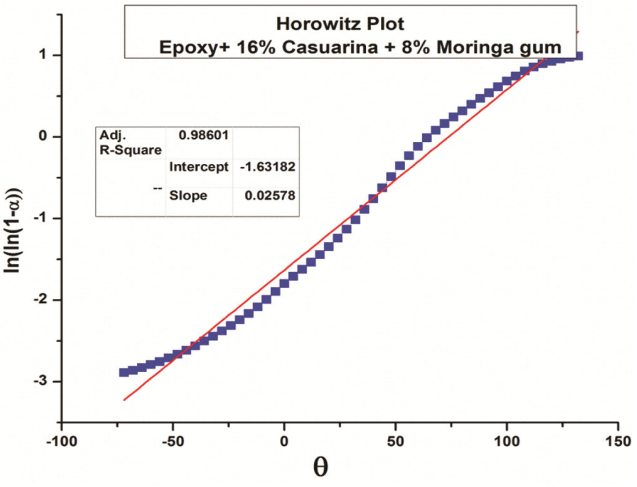

Fig. 13 — Horowitz plot of 8% Moringa gum + 16% Casuarina epoxy composite

Table 3 — Activation energy (E_a) and frequency factor (A) of Moringa gum + casuarina fibre Epoxy composite

Code	Epoxy resin	Epoxy + 16% casuarina leaf + 8% moringa gum
D1	Ea=54 $A=1.23X10^3$ $R^2=0.9310$	Ea=112.96 $A=1.77X10^7$ $R^2=0.9757$
D3	Ea=73.19 $A=1.62X10^4$ $R^2=0.9250$	Ea=126.09 $A=3.03X10^7$ $R^2=0.9717$
D4	Ea=65.53 $A=2.46X10^3$ $R^2=0.9289$	Ea=133.89 $A=1.53X10^8$ $R^2=0.9676$
F1	Ea=38.15 $A=1.63X10^1$ $R^2=0.89747$	Ea=67.14 $A=1.4X10^4$ $R^2=0.9529$
F2	Ea=64.58 $A=1.19X10^5$ $R^2=0.88294$	Ea=90.09 $A=1.83X10^6$ $R^2=0.9082$
R2	Ea=28.89 $A=0.72X10^1$ $R^2=0.90665$	Ea=58.02 $A=1.01X10^3$ $R^2=0.9655$
R3	Ea=31.69 $A=1.01X10^1$ $R^2=0.90512$	Ea=60.88 $A=1.25X10^3$ $R^2=0.9622$
MPL $^{1/3}$	Ea=31.69 $A=3.03X10^1$ $R^2=0.90512$	Ea=60.88 $A=3.74X10^3$ $R^2=0.9622$
MPL $^{1/2}$	Ea=28.89 $A=1.45X10^1$ $R^2=0.90665$	Ea=58.02 $A=2.01X10^3$ $R^2=0.9655$
MPL $^{2/3}$	Ea=26.39 $A=0.74X10^1$ $R^2=0.90658$	Ea=55.32 $A=1.12X10^3$ $R^2=0.9679$
A2	Ea=38.15 $A=8.14X10^1$ $R^2=0.89747$	Ea=67.14 $A=7.17X10^3$ $R^2=0.9529$
A3	Ea=38.15 $A=5.4X10^1$ $R^2=0.89747$	Ea=67.14 $A=4.78X10^3$ $R^2=0.9529$
A4	Ea=38.15 $A=4.07X10^1$ $R^2=0.89747$	Ea=67.14 $A=3.58X10^3$ $R^2=0.9529$
Broido model	Ea=58.17 $A=3.2X10^5$ $R^2=0.9504$	Ea=75.86 $A=2.3X10^5$ $R^2=0.9769$
Horowitz Model	Ea=67.56 $A=1.06X10^5$ $R^2=0.9566$	Ea=85.91 $A=2.03X10^4$ $R^2=0.986$

Table 4 — Thermodynamic parameters of 16% casuarina+8% moringa gum epoxy composite

Parameters	16% casuarina leaf + 8% moringa gum epoxy composite
Ea	112.96 kJ/mol
A	$1.77x10^7$
ΔH	107.69 kJ/mol
ΔS	-112.42 J/mol
ΔG	178.86 kJ/mol

activation energy of the hybrid composite (112.96 kJ/mol) compared to the epoxy–moringa gum (89.73 kJ/mol) and epoxy-casuarina (107.54 kJ/mol) composites reflects its superior thermal stability and improved energy resistance to degradation[46,47]. Therefore, the hybridization of moringa gum filler and casuarina fibre creates sustainable composites which prove to be beneficial in applications requiring high thermal stability.

Biodegradability test

The clean epoxy demonstrated negligible variation, with a slight increase to +0.12% observed at the 10-day mark, followed by a gradual decline to -0.70% at the 120-day interval. The observed minor overall shift aligns with the chemical inertness of plain epoxy, which is defined by a highly cross-linked network, limited accessible functional groups and an absence of biodegradable components. Consequently, the resistance of the neat epoxy to microbial degradation and moisture-induced swelling illustrates the durability of traditional thermoset polymers when subjected to soil conditions.

Nevertheless, the incorporation of biodegradable materials such as moringa gum and casuarina fibres significantly influenced the trajectories of weight change. The composite exhibited a gradual increase in weight initially attaining a value of +1.55% by day 20 (Fig. 14). However, a significant decline was observed, culminating in a reduction of -9.01% by the conclusion of the testing phase at the 120-day mark. The combined effects of hydrophilic polysaccharides in moringa gum and the cellulose-rich casuarina fibres enhance moisture uptake and create favorable conditions for microbial activity. These natural components, through enzymatic hydrolysis and microbial colonization, contribute to the progressive breakdown of the composite material over time. Consequently, the hybrid composite exhibits notable

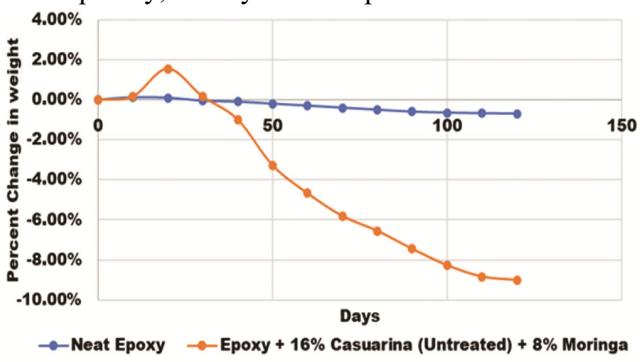

Fig. 14 — Biodegradability plot of composite

biodegradability, underscoring its potential as an eco-friendly material for sustainable applications where conventional synthetic materials pose disposal challenges[49,50].

Conclusion

The hybrid composite demonstrated a tensile strength of 38.5 MPa, with optimal flexural strength (53.5 MPa) and impact resistance (3074 J/m²) at 8% moringa gum, reflecting improved filler–matrix bonding and a balanced combination of toughness and stiffness. The maximum storage modulus is attained at a concentration of 8% moringa gum, as indicated by dynamic mechanical analysis measurements, which further reveal optimal stiffness and constraints on chain mobility. Thermogravimetric analysis revealed improved char formation and thermal resistance in the hybrid composite, with a residual mass of 15.5% compared to 2.77% for unreinforced epoxy resin, alongside a significantly higher glass transition temperature of 78.5 °C versus 59°C for the epoxy resin. Tmax was recorded at 360.1°C, significantly higher than those of epoxy resin, Epoxy moringa gum composite and Epoxy casuarina composite. This indicates superior thermal stability of hybrid composite compared to the individual fibre and filler composite. This analysis also revealed elevation in the activation energy of 112.96 kJ/mol, which indicates an improvement in thermal stability. It also enhances biodegradability, thereby rendering the composite suitable for environmentally sustainable applications. With an optimal composition that balances mechanical strength, thermal performance and biodegradability, epoxy bio-composites offer a promising solution for sustainable applications by combining the strength and biocompatibility of epoxy resins with the sustainability of bio-based materials.

Conflict of Interest

The authors declare that they have no conflict of interest.

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