Mechanical and Thermo-Mechanical Behaviors of Cork Filler-Reinforced Epoxy Composite

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Mechanical characteristics were studied for epoxy composites reinforced with cork fillers, analyzing various loading conditions of fillers ranging from 0 to 30%. The fabrication utilized a hand layup method. The results indicated that the composite mechanical properties, glass transition temperature, and storage modulus were optimal at a 20% natural filler loading. Dynamic mechanical analysis (DMA) showcased exceptional energy absorption capabilities up to 110 °C. Thermogravimetric analysis (TGA) showed that the bio-filler degraded quickly, leaving 0.3% remnant, but the cork filler composite (25% v/v) showed an even residue concentration of 9%. Additionally, biodegradability tests showed weight loss in a soil burial test with the addition of bio-filler to the composite.

DOI: 10.15376/biores.20.3.7540-7554

Keywords: Cork filler; Epoxy resin; Mechanical properties; Composite; Storage modulus; Framework

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INTRODUCTION

Bio-based plastic goods and biodegradable polymers, derived from renewable resources, offer promising alternatives for environmentally friendly and sustainable products, capable of competing effectively in today's market, as has been demonstrated (Mohanty *et al.* 2002; Satyanarayana *et al.* 2009; Fernandes *et al.* 2013). The investigations have examined the influence of coconut shell particles (CSP) on the thermal and mechanical properties of PLA biocomposites. The addition of raw as well as treated coconut hull powder reduced tensile strength but increased tensile modulus; however, treating the composite with a silane coupling agent improved tensile strength (Chun *et al.* 2012; Sareena *et al.* 2012; Irawan *et al.* 2022).

Composite materials have been produced using various reinforcing particles in vinyl ester resin, including sisal fiber, coconut shell powder, and combination of both. The influence of drilling parameters on thrust energy and torque has been studied (Venkatachalam *et al.* 2016). The manufacturing conditions, feed, cutting speed, and tool geometry were found to affect thrust energy and torque. Sisal fiber-reinforced composite exhibited the highest thrust and torque among all composites tested (Venkatachalam *et al.* 2016). Using snail shell particles as filler material in a polypropylene matrix, composites were prepared with varying filler content. Experimental results showed improvement in tensile modulus, flexural, and impact strength of the polypropylene composite with increasing filler content, compared to a reference talc filler material of 0.15 µm particle size (Onuegbu and Igwe 2011).

Banyan tree sawdust has been utilized as a filler to improve the mechanical qualities of polypropylene green composites. The inclusion of filler material affected abrasive water jet machining, with the impact of coupling agents and mineral filler material on machining qualities reported (Ramesha *et al*. 2016). The sawdust from the teak wood was used as filler material in high density polyethylene composites, which were prepared by using sawdust in various amounts (10, 20, 30, 40, 50, 60, and 70%). The effect of volume fraction on the mechanical properties was reported. The results showed that a filler content of more than 30% reduced impact strength (Bootkul *et al.* 2017).

Phenolic resin was used as matrix material, and sawdust and rice husk were used as reinforcements in powder form. The matrix filler ratio was taken as 60:40. Composites were prepared by using sawdust powder, rice husk powder, and both powders together. The experimental analysis examined the effect of ultraviolet light exposure on the water stability and mechanical properties of rice husk and sawdust composites. The results showed that phenol resin composites with sawdust had better mechanical properties than rice husk composites (Lette *et al*. 2018). Sawdust was reported to have better adhesion in the phenolic resin than the particulate rice husk filler material. The results showed that the composite with 6% sawdust powder resulted in better mechanical performance (Haliza Jaya *et al*. 2018).

A hybrid composite was prepared by adding different compositions of fly ash and sawdust. The impact of filler loading on the hardness, compressive strength, and moisture absorption was investigated. The results indicated that with a filler loading over the threshold level, the properties started to decrease due to poor wetting of the reinforcements by epoxy resin (Krishna *et al.* 2018). Composites were developed by using different volume fractions of sawdust and woven jute fibre in the epoxy matrix. The composite with three layers of jute fibre and 20% treated sawdust produced better values of mode 1 fracture toughness (Suthan *et al*. 2019).

In another study (Hiremath *et al.* 2018), the composite materials were fabricated by using egg shell particles as a filler material with glass fibre in polymer matrix. The hand layup method was used to create the composite material. Mechanical and physical properties were studied. The outcomes revealed that the stiffness can be improved by using a filler level of 10%. Composites were formulated using NaOH-treated and raw jute fabric to investigate the impact of nano-clay and eggshell powder. Ganesan *et al.* (2018) reported that composites containing 1.5% nano-clay and 1.5% eggshell powder exhibited superior mechanical and thermal properties. The study also demonstrated that NaOH chemical treatment significantly improved the mechanical properties of the composites and fostered good interfacial adhesion between the fibers and matrix, as evidenced by enhanced tensile and impact strength.

Composite films were fabricated from polylactic acid (PLA) and eggshell powder in varying percentages, as reported in a study by Ashok *et al.* (2014). Tests including X-ray, tensile, and micrograph analysis were conducted. Incorporating up to 4% eggshell powder enhanced tensile strength and modulus while also improving composite crystallinity. SEM images revealed uniform particle distribution at 4% loading, with higher concentrations leading to particle agglomeration. The addition of eggshell powder also improved the thermal stability of the PLA composite film (Ashok *et al.* 2014). Composites of eggshell powder and biodegradable thermoplastic starch exhibited properties akin to commercial calcium carbonate, with faster degradation. Eggshell inclusion enhanced thermal stability and moisture resistance. Filler particles, influenced by volume fraction, size, and matrix interaction, significantly impact thermal and mechanical properties, improving surface characteristics such as hardness and tribological traits (Bootklad and Kaewtatip 2013). The optimal range for enhancing mechanical properties in epoxy composites is typically achieved by incorporating natural fibers or fillers at levels between 20% and 25%, as indicated by Parthasarathy *et al.* (2023, 2024).

Natural and glass fibers reinforced an epoxy matrix, enhancing mechanical and wear properties. Alkali-treated areca and glass fibers (20 wt% each) achieved great tensile strength , with improvements in flexural strength, impact resistance, and reduced wear loss observed at higher fiber content, aided by better fiber-matrix adhesion (Sumesh *et al.* 2024). NaOH-treated areca nut husk fibers in epoxy composites achieve peak strength and wear resistance at 20 wt% fiber content, but remain less competitive than other natural fibers, suggesting room for process improvements (Palanisamy *et al.* 2024). Hybrid ramie/flax fibers in epoxy resin composites showed improved mechanical strength with optimal fiber content, length, and controlled alkali treatment. SEM analysis confirmed better fiber distribution, though excessive fabrication conditions reduced performance (Sumesh *et al.* 2023).

Asparagus bean stem fiber (ABSF) has been used as reinforcement in polymer composites (Jiratti *et al.* 2024). Results showed that alkali-treated ABSF significantly improved mechanical properties due to enhanced fiber-matrix bonding. Results indicate ABSF's potential as a viable, eco-friendly alternative for lightweight structural applications (Jiratti *et al.* 2024). Recycled manhole cover composite (CMC) flour and MDF sawdust have been investigated as low-cost fillers in recycled polypropylene (PP) composites. The CMC flour, obtained from ground recycled composite materials used in manhole cover production, together with MDF sawdust, improved the bending strength and modulus of the composites with minimal impact on water absorption and thermal stability. These findings demonstrate a sustainable approach to producing strong, water-resistant PP composites (Ayrilmis *et al*. 2024).

That ultrasonic silane treatment of areca fibers in tri-layer epoxy composites enhanced mechanical properties, with notable improvements in tensile strength, hardness, flexural strength, and impact resistance. Morphological analysis confirmed strong fiber-matrix bonding, highlighting the effectiveness of surface modification and stacking sequences in optimizing composite performance (Jafrey *et al.* 2024). Developing brake friction composites from untreated and surface-processed biofiber derived from *Cardiospermum halicababum* agro waste. The study found that silane-treated fibers offered a higher friction coefficient than untreated and alkali-treated fibers, with distinctive wear patterns observed in SEM analysis, underscoring their potential as sustainable alternatives to commercial brake pads (Vijay *et al.* 2024). The novel aerial root fiber of *Ficus retusa* L. (FRL) for its potential as a reinforcement in natural fiber-reinforced composites (NFRC). Findings revealed FRL's high cellulose content, crystallinity, tensile strength, and thermal stability, indicating excellent bonding and mechanical properties, making it a promising eco-friendly alternative to synthetic fibers (Palaniappan *et al.* 2024).

This research focuses on examining the effects of incorporating cork powder material into an epoxy matrix as a fiber material, aiming to evaluate its influence on the thermal properties, tribological, mechanical, vibration, and dynamic mechanical of epoxy composites. The study specifically investigated its potential suitability for small and medium-scale applications. While previous studies have commonly utilized a variety of bio and non-fillers/fibers in epoxy composites, the novelty in this research involves finding the ideal value for the formulation of a composite utilizing cork filler and epoxy in various percentages.

Bio-based composites offer an eco-friendly alternative to plastics as they are made from renewable resources such as plant fibers. They help reduce reliance on fossil fuels and decrease carbon emissions, contributing to a more sustainable future. Additionally, biocomposites often biodegrade more easily than traditional plastics, mitigating environmental pollution.

EXPERIMENTAL

Cork filler blended with epoxy resin was used in this study, with the workflow shown in Fig. 1.

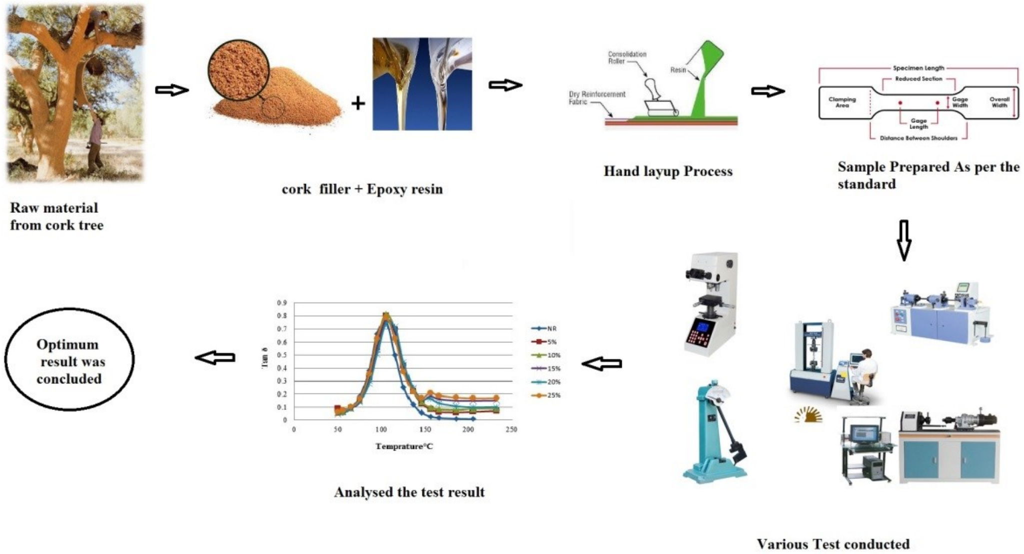

Fig. 1. Work flow of cork filler epoxy composite study

The density of the cork filler was calculated and found to be 1.06 g/cm^3. Over the course of 4 h, the natural filler material was ground to a finer particle size of 5 to 10 microns using a ball mill. Then, it was blended with various percentages of cork filler-blend epoxy (5, 10, 15, 20, 25, and 30% by volume). To achieve uniform dispersion, a mechanical mixer was used to continually swirl both the cork powder and epoxy resins for 45 minutes. The hand layup method was used to create cork filler with epoxy polymer material laminates as shown in Fig. 2. The laminates were snipped for various tests within a week of curing in accordance with ASTM guidelines.

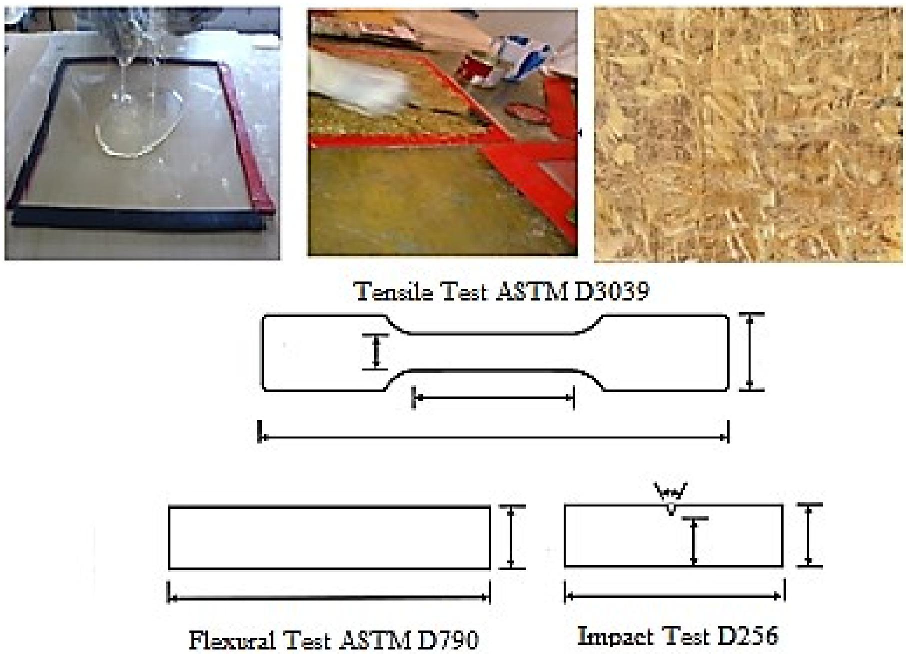

Fig. 2. Sample preparation was conducted using the hand layup method

Mechanical Properties

The Instron universal testing machine was employed for tensile and flexural testing. Following ASTM D3039 (2017) standards, the tensile test was conducted at a speed of 2 mm/min. Flexural strength was assessed through a three-point bending test, adhering to the procedures outlined in ASTM D790 (2017). Impact strength was determined using the Izod method, following the guidelines specified in ASTM D256 (2023) as shown in Table 2. Each test utilized five samples, and the mean results were calculated. Table 1 shows the specimen compositions.

Table 1. Composition of Specimens

Sample No	1	2	3	4	5	6	7
Epoxy (%)	100	95	90	85	80	75	70
Cork (%)	0	5	10	15	20	25	30

Table 2. Specifications and Dimensions for the Test Specimens

S. No	Testing Methodology	Standards	Dimension (mm)
1	Tensile Testing	ASTM D3039	250 x 25 x 2.5
2	Flexural Testing	ASTM D790	154 x 13 x 3
3	Impact Testing (Izod)	ASTM D256	63.5 x 12.5 x 3.2

Dynamic Mechanical Analysis

Using the SEIKODMAI-DMSC 6100 instrument from Seiko Instruments Inc., Japan, the epoxy polymer produced from cork filler was investigated under high and dynamic stress conditions. The temperature ranged from 40 to 250 °C and was employed for dynamic mechanical analysis (DMA). The testing protocol included 10 Hz as the frequency and 5 °C/min as the average heating rate.

Thermogravimetric Analysis (TGA)

The composite specimens were evaluated for thermal stability using the TG/DTA 6200 SEIKO TGA analysis tool from Seiko Instruments Inc., Japan. Spanning the temperature that range from 0 to 800 °C, the sample was heated in a nitrogen environment to avoid oxidation during the analysis, which entailed a temperature increment of 20°C/min.

Fractography Study

Morphological analyses were performed by a scanning electron microscope (SEM) to assess the filler particle's diffusion in the polymer matrix. For the experiment, a Hitachi S3400 N scanning electron microscope was utilized. To avoid charging, the composite specimens were plated through with gold using sputtering.

Biodegradability Test

A common and widely used technique for assessing deterioration is soil burial, which makes use of the dynamic ecosystem that is produced by various species under waste disposal settings. Biodegradability of the sample was gauged through continual monitoring of weight reduction in the soil. Initial weighing preceded burial, and retrieval occurred at 10-day intervals up to 120 days. After extraction, rinsing and reweighing were conducted to quantify weight loss.

RESULTS AND DISCUSSION

This work considered the integration of cork filler blended with epoxy resin to enhance the characteristics of biopolymer materials. A comprehensive investigation was conducted to compare this outcome with the properties of epoxy resin alone. Figures 3, 4, and 5 illustrate the mechanical capabilities of cork filler blended with epoxy resin qualities of epoxy polymer composites.

The outcomes of the tensile study performed on a variety of composite samples are shown in Fig. 3. This research examined the modulus and total tensile strength of various composite sample combinations. The composition consisting of twenty percent cork powder and eighty percent epoxy resin demonstrated the highest performance, with a total tensile strength of 37 MPa and a modulus of 1240 MPa. The results underscore the significant influence of the filler volume ratio on the composite's tensile characteristics.

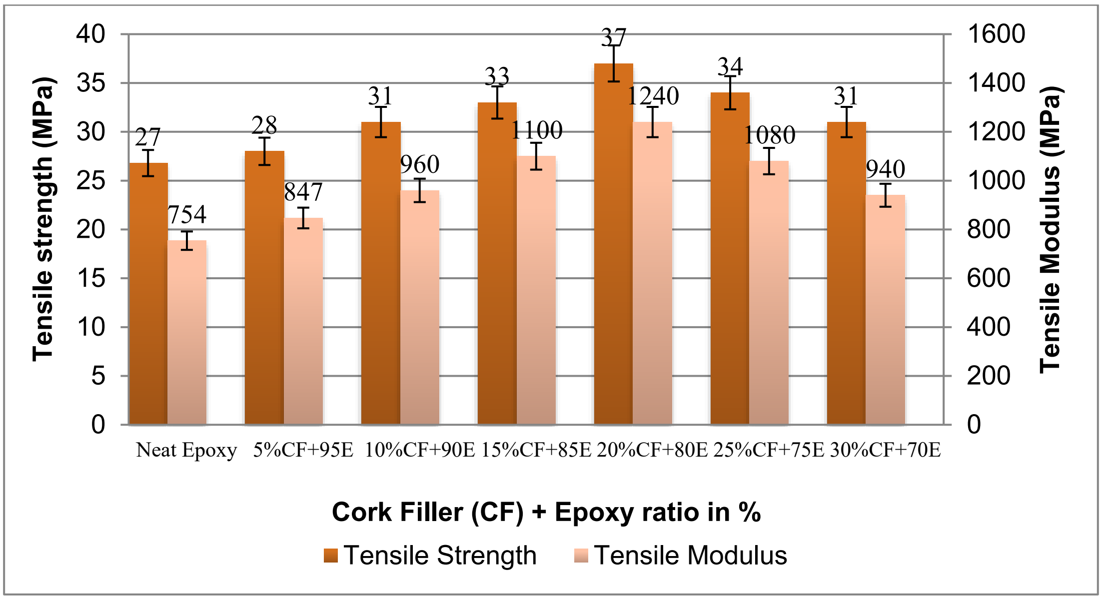

Fig. 3. Tensile characteristics of neat epoxy and epoxy loaded with cork filler

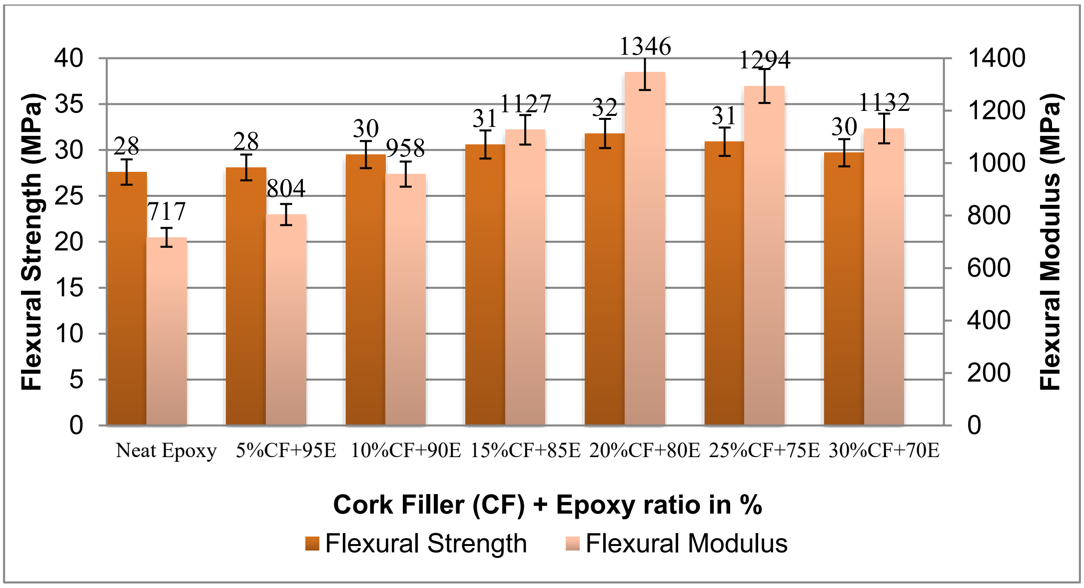

Fig. 4. Flexural characteristics of neat epoxy and epoxy loaded with cork filler

The findings emphasize the necessity of fine-tuning the composition, especially the filler volume ratio, to improve the mechanical properties of the composite material. Subsequent discussion will explore the observed relationships and correlations among filler properties, mechanical performance and volume ratios across the parameter spectrum investigated.

The outcomes of the flexural study performed on a variety of composite samples are shown in Fig. 4. The graph shows how different compositions varied in terms of flexural strength and modulus. More specifically, the composition with 80% epoxy resin and 20% cork powder exhibited the highest modulus and flexural strength, measuring 32 and 1346 MPa, respectively. It is interesting to note that the modulus and flexural strength of the composite material both clearly decline with increasing filler material integration. Thus, the fibre content affects the composite flexural characteristics.

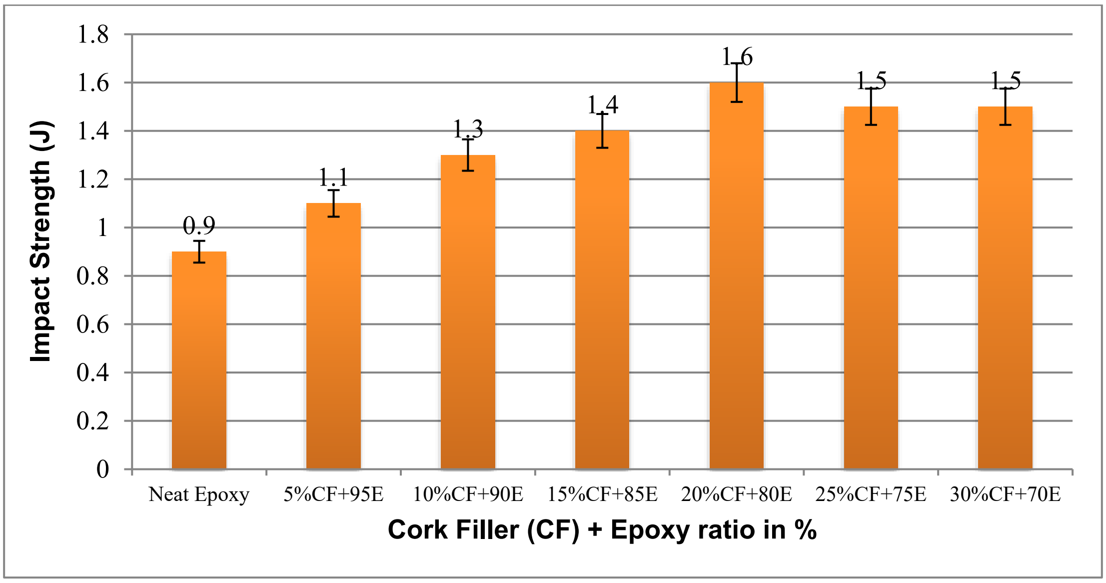

Fig. 5. Impact properties of cork filler blended epoxy with epoxy blender hybrid material

The outcomes of the impact strength study performed on a variety of composite samples are shown in Fig. 5. Additionally, the graph demonstrates that composition with 80% epoxy resin and 20% neat cork filler had the maximum impact strength, measuring 1.6 J. Impact strength measures the ability of material to endure intense and sudden loads for a brief period of time (Venkateshwaran *et al*. 2011). The impact testing process subjects materials to significant strain in a brief period of time; hence it is predicted that there will be minimal variances in impact strength values among the examined compositions. The consequences of these impact vigor quantifications for the overall effectiveness and applicability of the composite material will be further discussed.

The 20 to 25% reinforcement level in bio-composite materials typically yielded optimal results by enhancing strength and stiffness without excessive brittleness. Particularly at the 20% level, reinforcement particles were well-distributed, improving bonding and preventing weak spots. This balance also maintains a lightweight profile, making it suitable for applications needing both strength and reduced weight.

Dynamic Mechanical Analysis

Storage modulus (E′)

Figure 6 shows how temperature and frequency affected a composite material's storage modulus (*E′*), which is made up of 20% filler and epoxy resin. Between 40 and 250

°C was the test temperature range, and the frequency was 10 Hz. According to the findings, the composite showed a better storage modulus at any frequency up to 110 °C. Both the resin and the composite showed almost the same energy utilization as the testing temperature rose. This implies that the composite material successfully supported the load up to 110 °C (George *et al*. 1996). Nevertheless, a steady loss of grip on the matrix may occur above this temperature, potentially impairing their ability to perform as planned.

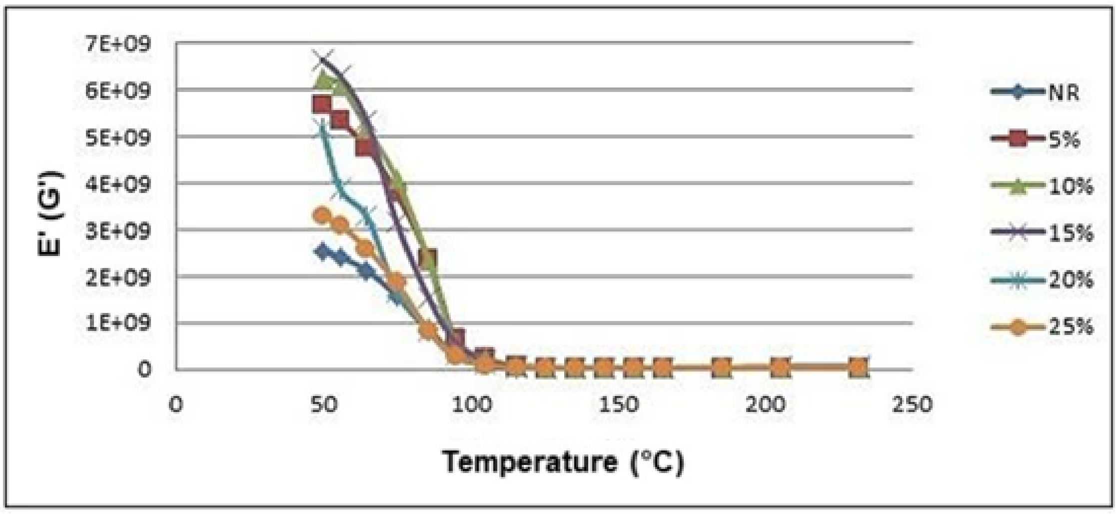

Fig. 6. Storage modulus of epoxy resin and cork filler composite at 10 Hz frequency

Loss factor (tan δ)

A common way to express the loss factor (tan δ) is as the ratio of the loss modulus (G'') to the storage modulus (G'), that is, $\tan\delta = G'' / G'$. This ratio reflects the balance between energy dissipated as heat and energy stored elastically during deformation, and it provides insight into the molecular mobility within the polymer structure (Chandramohan *et al.* 2024).

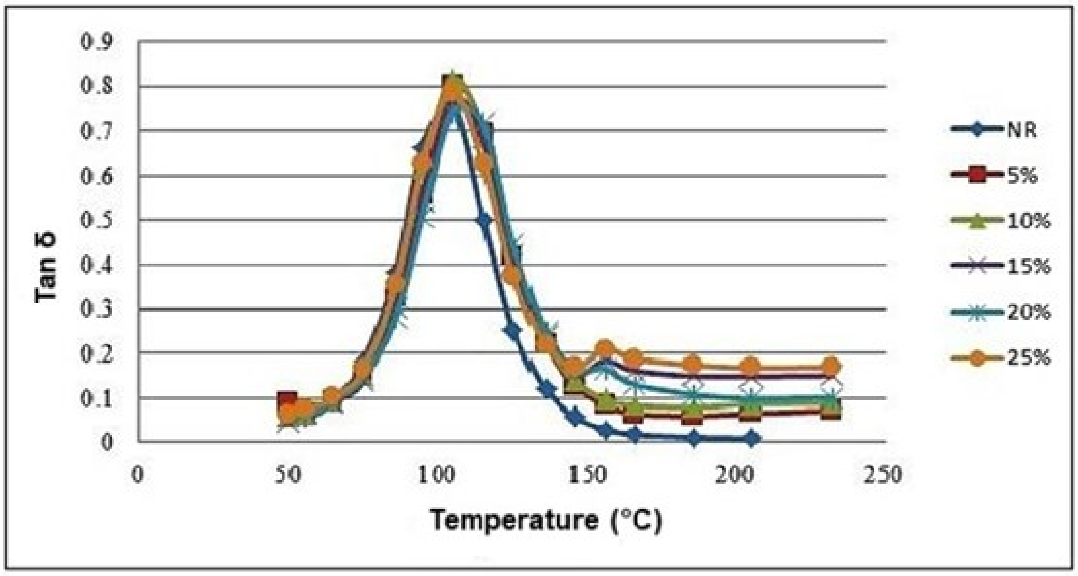

Fig. 7. Storage modulus of epoxy resin and cork filler composite at 10 Hz frequency

The data indicate that adding reinforcement reduces the loss component of the composite, suggesting that the presence of reinforcement alters the energy dissipation mechanism compared to the pure polymer matrix. At lower temperatures, the material exists in a glassy state, characterized by limited molecular mobility. As the temperature increases and reaches the glass transition temperature (T_g), the material undergoes a phase transition. Above T_g, the polymer enters the rubbery or elastic state, where increased molecular mobility allows for greater segmental motion and flexibility.

It is common to correlate the maximum loss modulus (E'' max.) or the highest loss factor (tan max.) with the temperature at which this transition takes place. In this study, the greatest tan value is used to calculate the glass transition temperature. Glass transition temperatures (T_g) shifted significantly as applied frequency was increased, as evidenced by the tan curve's decreasing peak when compared to epoxy resin. According to this shift in T_g values, the filler working to promote change is likely a contributing factor.

As the material transitions from a rigid to a more flexible state, the glass transition region is associated with the damping (tan δ) peak. With increasing temperature, the initial stability of the filler particles influences the polymer structure, allowing molecular segments to form small clusters and chain-like configurations. Consequently, the degree of molecular mobility within the heterogeneous phases increases, leading to a rise in the tan δ peak.

Thermogravimetric analysis

Figure 8 illustrates the TGA curves for both neat epoxy resin and the hybrid polymer material containing cork. The epoxy polymer material, composed of pure epoxy with added cork-blended epoxy resin, exhibited higher thermal stability compared to neat epoxy resin. The results indicate that the incorporation of cork-blended epoxy material has no discernible impact on thermal stability. However, the cork-blended epoxy resin components contributed to a slight enhancement in TGA values.

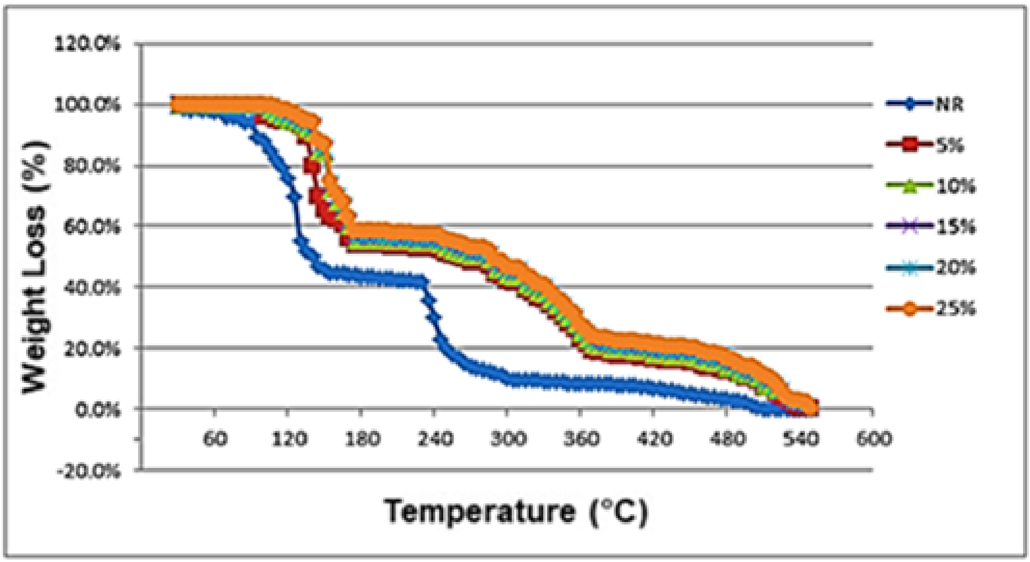

Fig. 8. TGA curves of epoxy resin and cork filler reinforced epoxy composites

The initial weight reduction that was noticed was ascribed to water evaporation. Thus, the thermal stability of the polymer material was enhanced by the addition of epoxy resin that was blended with cork to the epoxy matrix. The epoxy resin was stable up to 491 °C, as shown in Fig. 7, after which it dissolved quickly and left only 0.3% . As an illustration of its improved thermal performance, the cork-blended epoxy displayed stability up to 25% and with a residual content of 9%.

The SEM image in Fig. 9 revealed hybrid polymer materials with 20% natural resin added. The natural resin was evenly distributed throughout. Because of this equal dispersion, the load-carrying capacity of hybrid polymer composites was improved. The SEM of 30% hybrid polymer material is shown in Fig. 10. The addition of natural resin resulted in agglomeration. Agglomeration reduced the effective surface area available for interaction between the filler particles and the matrix, limiting the extent of interfacial bonding and thereby reducing the composite’s mechanical strength, stiffness, and toughness. It also creates inconsistencies in load distribution and material properties, impacting overall performance. By addressing agglomeration, these issues can be mitigated, leading to enhanced durability and more reliable composite performance.

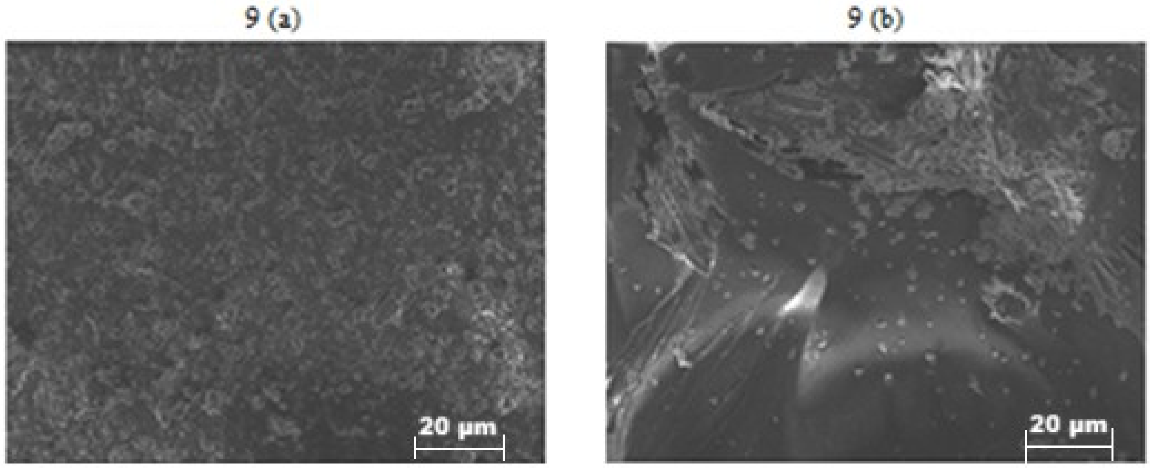

Fig. 9. SEM images of 20% cork filler blended epoxy with epoxy resin showing even dispersion for 9(a) surface area, 9(b) cross-sectional area

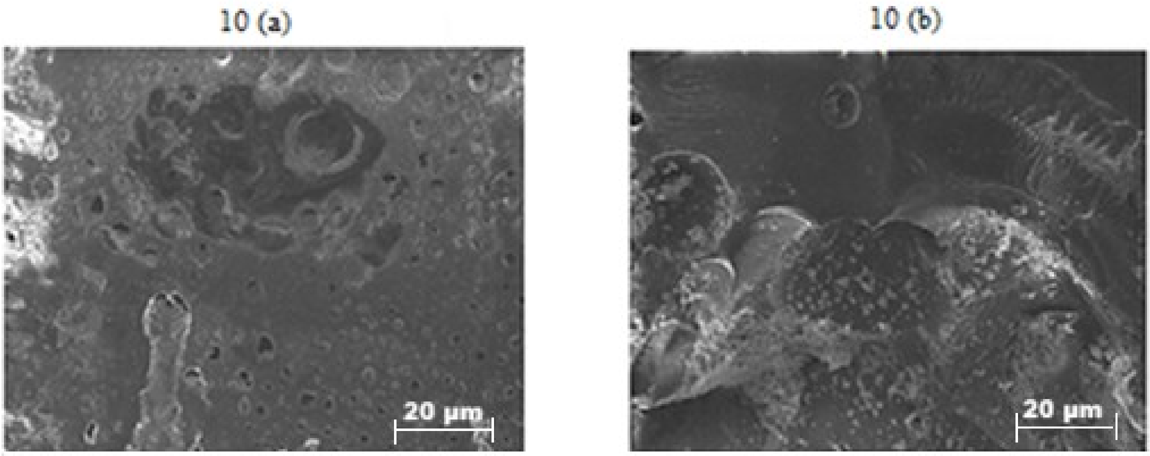

Fig. 10. SEM images of 30% cork filler blended epoxy with epoxy resin agglomeration for 10(a) surface area, 10 (b) cross-sectional area

To prevent agglomeration and to improve composite performance, coupling agents can enhance filler-matrix bonding, while optimizing filler concentration ensures better dispersion. Advanced mixing techniques and filler surface modifications, such as chemical treatments, also help reduce clumping. Controlling curing and processing conditions further minimizes agglomeration, ensuring uniform composite properties.

Biodegradability test

Biodegradability testing involved burying a material sample in damp soil, as illustrated in Fig. 11, to assess the impact on cork-blended epoxy polymer materials. Before burial, the sample's initial weight was recorded, and further measurements were collected at regular intervals to track any changes. After 120 days, the samples exhibited a loss of approximately 5% of their total weight, as shown in Fig. 12. Initially, the sample weight increased due to moisture absorption but then started to decrease.

Fig. 11. Biodegradability test for cork-epoxy composite

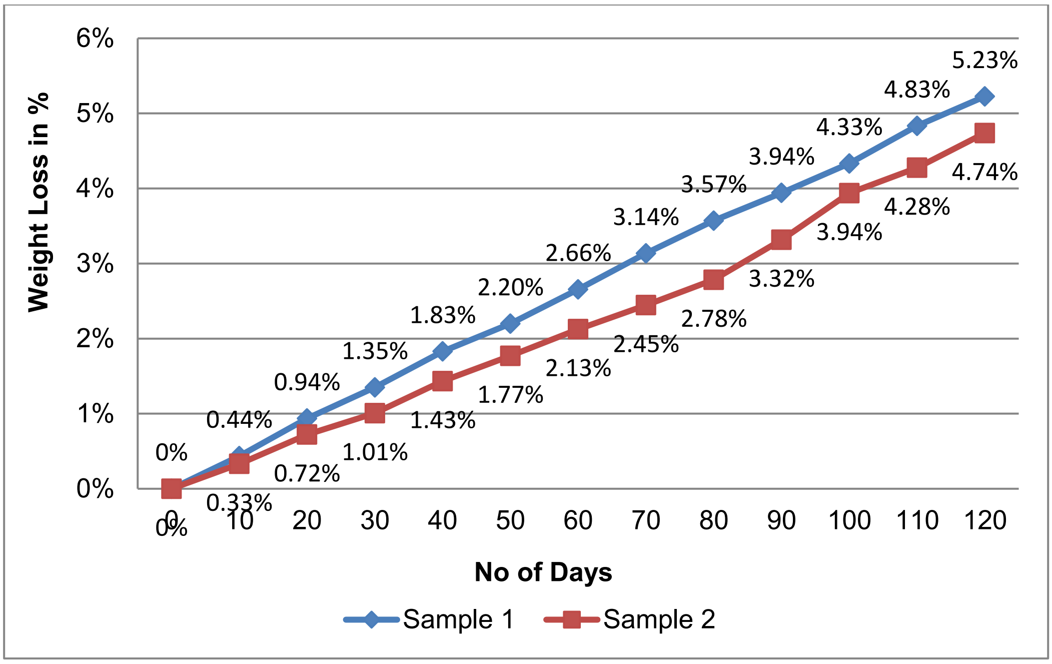

Fig. 12. Biodegradability curves of epoxy composite reinforced with cork filler

The interaction with micro- and macro-organisms was attributed to the weight loss observed in the composite sample over time. A comparative evaluation of the material with other natural fiber and filler-reinforced polymer composites showed that its biodegradability was higher. Based on various research and literature (Parthasarathy *et al.* 2023, 2024), the addition of bio-based fillers increases the degradability of composite materials, and the incorporation of bio-based fillers in epoxy hybrid materials contributes to the production of more environmentally friendly polymers.

Bio-composite materials have limitations such as lower strength, moisture sensitivity, and reduced thermal stability, which affect durability and performance in demanding applications. Additionally, natural fiber variability and processing challenges can lead to inconsistent quality. Future work is being considered to increase bio-filler content in composites without affecting quality. It is planned to optimize filler size and distribution for uniformity, as well as to use coupling agents to improve filler-matrix bonding. Pre-treating fillers and adopting hybrid filler systems with synthetic materials can also help maintain performance. Strengthening the matrix material and controlling processing conditions ensures better compatibility with higher filler content. These strategies may allow for increased sustainability while preserving composite properties.

CONCLUSIONS

The influence of cork filler on the mechanical characteristics of epoxy resin was examined in this study. Cork wood powder was employed in the formulation of many epoxy composites as a bio-filler.

1. A thorough analysis of the mechanical characteristics revealed enhancements in the impact, tensile, and flexural strengths of the composite containing 20% v/v bio-filler.
2. The integration of the bio-filler significantly altered the tensile and flexural moduli, indicating increased stiffness in the composite.
3. With a volume proportion of 20% cork powder filler and 80% epoxy, the filler and resin's strong compatibility allowed for the optimization of the outcomes.
4. Thermogravimetric analysis showed that the filler degraded quickly, leaving 0.3% of residue behind. However, the 25% v/v cork filler composite showed a continuously steady 9% residue content.
5. Biodegradability assessments demonstrated that incorporating a bio-filler into the composite led to a reduction in weight during a soil burial test.
6. The heightened rigidity of composite materials is crucial in industries prioritizing mechanical strength. In aerospace, stiffer epoxy composites reduce weight, improving structural performance and fuel efficiency.

ACKNOWLEDGEMENT

The authors acknowledge the funding from the Ongoing Research Funding Program (ORF-2025-355), King Saud University, Riyadh, Saudi Arabia.

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Article submitted: September 2, 2024; Peer review completed: October 19, 2024; Revised version received: December 3, 2024; Revisions accepted: July 13, 2025; Published: July 24, 2025.
DOI: 10.15376/biores.20.3.7540-7554