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Thermal Effects on Compression Behavior and Microstructure of Wheat and Rice Fiber-Reinforced Polymer Hybrid Composite Bricks for Civil Infrastructure

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ABSTRACT

The environmental impact of conventional construction materials has driven the need for sustainable alternative in civil infrastructure. In current times, natural fibers-based composites have increasingly used as non-metallic reinforcement for new construction and for strengthening of structures, making assessment of their mechanical and thermal properties, and performance essential. In this study, the compressive behavior of hybrid wheat and rice fiber-reinforced polymer (WRFRP) composites is assessed at low to moderate temperatures as encountered during typical environmental and service scenarios in structural systems. An alkali treatment is applied on wheat fibers before composite preparation. These composites are exposed to 40°C, 50°C, and 60°C prior to compressive behavior analysis and their performance is compared with that at room temperature (RT). Further, scanning electron microscopy (SEM) and differential scanning calorimetry (DSC) analyses are performed to investigate damage patterns, surface morphology, and thermal behavior at elevated temperatures. Natural fibers in these composites are treated in an alkaline solution to remove impurities and enhance the bond between fibers and binders. The peak average compressive strengths of WRFRP composites are reported as 21.5 MPa at RT, which reduces by 50% at 60°C. Moreover, empirical equations are proposed to estimate relations for linear stress–strain and peak compressive strength-temperature increment, respectively. The study helps understand the behavior of agricultural residue-based composites at low to moderate temperatures.

HIGHLIGHTS

- Novel hybrid wheat and rice fiber-reinforced polymer (WRFRP) composite development.
- Compressive behavior of WRFRP composites at low to moderate temperatures.
- Microstructural analysis composites of WRFRP at ambient, 40 °C, 50 °C, and 60 °C.
- Temperature dependent empirical models for thermal performance of WRFRP composites.
- Thermal exposure leads to fiber damage, voids, and interfacial debonding in WRFRP.

摘要

隨著人們對傳統建築材料環境影響的擔憂日益增加，土木基礎設施對替代性環保材料的需求也隨之提升。當前，以天然纖維為基礎的複合材料作為非金屬增強材料，在新建工程及結構加固領域的應用日益廣泛。因此，對此類生物複合材料進行力學與熱學特性分析，以及性能評估，至關重要。本研究針對混合小麥與稻米纖維強化聚合物 (WRFRP)複合材料，在結構系統中常見的典型環境與使用情境所涉及的低至中溫條件下，評估其壓縮行為。在分析其壓縮行為前，將這些複合材料分別置於40°C、50 °C 及 60 °C 環境中，並與室溫下的行為進行比較。此外，透過掃描電子顯微鏡 (SEM)與差示掃描量熱法 (DSC)分析，探究高溫下的損傷模式、表面形態及熱行為。這些複合材料中的天然纖維經鹼性溶液處理，以去除雜質並增強纖維與黏合劑之間的結合力研究指出，WRFRP複合材料的峰值平均抗壓強度為21.5 MPa，在60 °C時會分別降低50%。此外，本文提出分析方程式，分別用於估算線性應力-應變關係及峰值抗壓強度-溫度增量關係。根據掃描電子顯微鏡 (SEM)與差示掃描量熱法 (DSC)

KEYWORDS

Alkali treatment; biocomposite; compressive strength; hybrid polymer composite; nonmetallic reinforcement; rice fiber; thermal behavior; wheat fiber

關鍵詞

鹼處理; 生物複合材料; 抗壓強度; 混合聚合物複合材料; 非金屬增強材料; 稻米纖維; 熱行為; 小麥纖維

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分析，研究指出在低至中溫範圍內會出現纖維損傷、孔隙及界面脫黏現象，並分別釐清了熱轉變點。本研究有助於理解以農業殘餘物為基材的複合材料在低至中溫下的行為表現。

Introduction

The increasing impacts of environmental pollution and climate change have necessitated the need to find alternatives to conventional, high-carbonintensive construction materials. Similarly, using synthetic fiber composites in the construction sector also contributes to massive carbon emissions. On the other hand, many countries currently face challenges with agricultural waste management, often burning it, which leads to air pollution. Therefore, utilizing the most generated agricultural residues, such as wheat straw, rice husk, and bagasse, in the construction industry can help reduce greenhouse gas emissions due to the factors above (Jagtap et al. 2024).

In the last decade, natural fiber-reinforced polymer composites showed significant potential in civil infrastructure for structural applications, e.g., reinforcement, strengthening (Kushwaha, Bahurudeen, and Ajinkya 2025; Saini and Matsagar 2023), insulation boards, architectural applications, acoustics (Saleem, Khan, and Arumugam 2024; Saleem, Orra, and Arumugam 2023), and an alternative to wooden boards (Ahmad et al. 2022). In traditional structures, straw-reinforced cementitious, lime-based, and clay-based practices were commonly implemented. The most widely used natural fibers, e.g., banana, flax, jute, hemp, sisal, and wheat, used in the form of fabric, long fibers, short/chopped fibers, composite, and rebar are effectively utilized in civil infrastructure (Prajer and Ansell 2014; Saini, Matsagar, and Kodur 2024). Some researchers (Seile, Spurina, and Sinka 2022) showed that flax- and hemp-reinforced polylactic acid composites help reduce carbon emissions by providing an alternative to conventional materials and by helping accumulate carbon. The typical applications, environmental advantages, and high mechanical strength of Camellia oleifera shells, green coconut, and sugarcane bagasse fibers in particleboards have been reported by Fiorelli, Bueno, and Cabral (2019) and S. Yang et al. (2024).

In recent years, researchers have increasingly focused on identifying sustainable methods for utilizing agricultural residue-based fibers, i.e., wheat straw, rice husk, and sugar bagasse (Manikandan, Ramesh, and Saleem 2025). For effective utilization of these natural fiber-reinforced polymer (NFRP) composites for structural applications, a thorough understanding of their physical, mechanical, and thermal behavior is necessary. There are various strategies, such as including additives in the mix of composite, applying additional coatings, and providing treatment to natural fiber for enhancing the performance of NFRP composites. Ismail, Belayachi, and Hoxha (2020) reported the effective use of natural additives, e.g., hemoglobin, casein, and gelatin, for wheat straw in lime and gypsum plaster composites to enhance thermal conductivity and load-carrying capacity. More than 20% enhancement in tensile strength of rice and wheat straw-based composites was reported due to alkali treatment of the fibers by Q. Yang et al. (2023) and Shifa, Kanok, and Haque (2024), respectively. Moreover, improved thermal stability and mechanical strength of wheat straw-reinforced polymer composites were observed due to alkali and nano clay treatments by Mittal and Sinha (2017) and Naghdi and Nejat (2023), respectively. Hybridization of natural fibers with synthetic or natural fibers in polymer composites was also proven an effective strategy to significantly enhance the performance of natural fiber-based composites (Ali-Eldin et al. 2019; Fong et al. 2025; Saleem, Khan, and Arumugam 2024; Velmurugan et al. 2023). Also, this promotes applications in automobile, civil, textile, aerospace, and turbine industries. However, there is no study investigating the thermal effects on compressive behavior and surface morphology of novelly developed wheat and rice fiber-reinforced polymer (WRFRP) hybrid composite bricks. Therefore, in this study, the compressive behavior and microstructural degradation of WRFRP composite bricks are explored at different low and moderate temperatures. Assessing the thermal performance of biocomposites in the temperature range from room temperature to 60°C is crucial for structural applications, as these conditions commonly occur in service environments exposed to sunlight, heat waves, or industrial processes. Evaluating biocomposite behavior within this range ensures that the developed composites maintain mechanical integrity, dimensional stability, and durability

under realistic operating temperatures. Also, accordingly, some strengthening measures can be taken to enhance their reliability and lifespan in practical applications.

The main objectives of this study are: (a) to investigate the compressive behavior of novelly developed wheat and rice fiber-reinforced epoxy polymer (WRFRP) hybrid composites at room temperature (RT), (b) to investigate the effect of low to moderate temperatures, i.e., 40°C, 50°C, and 60°C on the compressive behavior of WRFRP and respective failure patterns, and (c) to examine the influence of low to moderate temperatures on the surface morphology and damage in WRFRP composites. The study contributes to understanding the adaptability and feasibility of WRFRP for structural applications exposed to different temperatures.

Production of wheat and rice fiber-reinforced polymer hybrid composite bricks

In this study, biocomposites have been developed using natural agricultural residues, specifically wheat straw and rice husk, as reinforcing fillers in an epoxy matrix. Agricultural residues are extracted from crops grown in the Uttar Pradesh region of India. Alkali treatment is applied to short wheat fibers (size, 5 mm to 15 mm) before mixing with the binder.

Processing of fibers

Pretreatment of natural fibers is essential to improve their compatibility with polymer matrices by removing surface impurities such as lignin, hemicellulose, waxes, and oils. This process enhances fiber–matrix adhesion, increases surface roughness, and improves the mechanical performance of the resulting composite material. Various pretreatment methods, alkali treatment (Mittal and Sinha 2017; Naghdi and Nejat 2023; Neudecker et al. 2025), thermal treatment (Pan, Zhang, and Dingguo 2009), acid treatment, enzymatic treatment, microwave-assisted treatment, plasma treatment, and silane treatment (Faruk et al. 2012), have been proven effective for wheat fibers. Among these, the present study employs alkali pretreatment using 1% sodium hydroxide (NaOH), which effectively eliminates unwanted components while preserving fiber integrity, thereby enhancing interfacial bonding and overall composite performance (Jittin, Bahurudeen, and Ajinkya 2020). The fibers (10% w/w) have been soaked for 24 h at room temperature. After alkali treatment, the fibers are thoroughly washed with water until the pH is neutralized to 7. Subsequently, the fibers are oven-dried to remove residual moisture and prepare them for composite fabrication.

Fabrication of WRFRP composite bricks

The wheat and rice fiber-reinforced polymer (WRFRP) hybrid composites have been developed using the hand lay-up and compression method, as shown in Figure 1. The composites consist of randomly oriented wheat short fibers, a rice husk powder serving as the reinforcing phase, and epoxy resin as the polymeric binder. The rice husk powder fills the air gaps between the wheat fibers, ensuring a smooth, homogeneous surface in the composite. A final composition of 20% treated wheat fiber, 50% rice husk powder, and 30% epoxy resin by weight has been selected for fabricating the hybrid composite. The ratio of each component is determined through several pretrials to maintain integrity, eliminate gaps, achieve a smoother surface, ease the chosen fabrication process, and enhance relative strength. Moreover, using additional rice husk powder helps reduce the binder content, achieving a uniform mix without air gaps. In addition, the objective of the study is to demonstrate the use of stubble (wheat and rice straw-based waste, generally burnt in many countries) in biocomposites with good compressive strength. The epoxy resin and hardener have been mixed in a 2:1 weight ratio, as recommended by the manufacturer, to ensure optimal curing performance and mechanical strength. Alkali treatment (e.g., with 1% NaOH solution) has been applied to wheat fibers to remove surface impurities, enhance surface roughness, and improve interfacial adhesion between fibers and the matrix (cf. processing of fibers).

The treated fibers were mixed into the epoxy mixture to ensure uniform coverage of the fiber surface with resin and to minimize visually apparent agglomeration. The homogeneous mixture is then cast into a steel mold with internal dimensions of 190 mm × 90 mm × 90 mm, equivalent to the size of a standard construction brick. The casting has been performed in five successive layers to improve composite quality, reduce the possibility of air entrapment, and ensure even fiber distribution across the composite. After the addition of each layer, manual compaction has been carried out using a heavy steel cube of dimensions similar to the inner dimensions of the

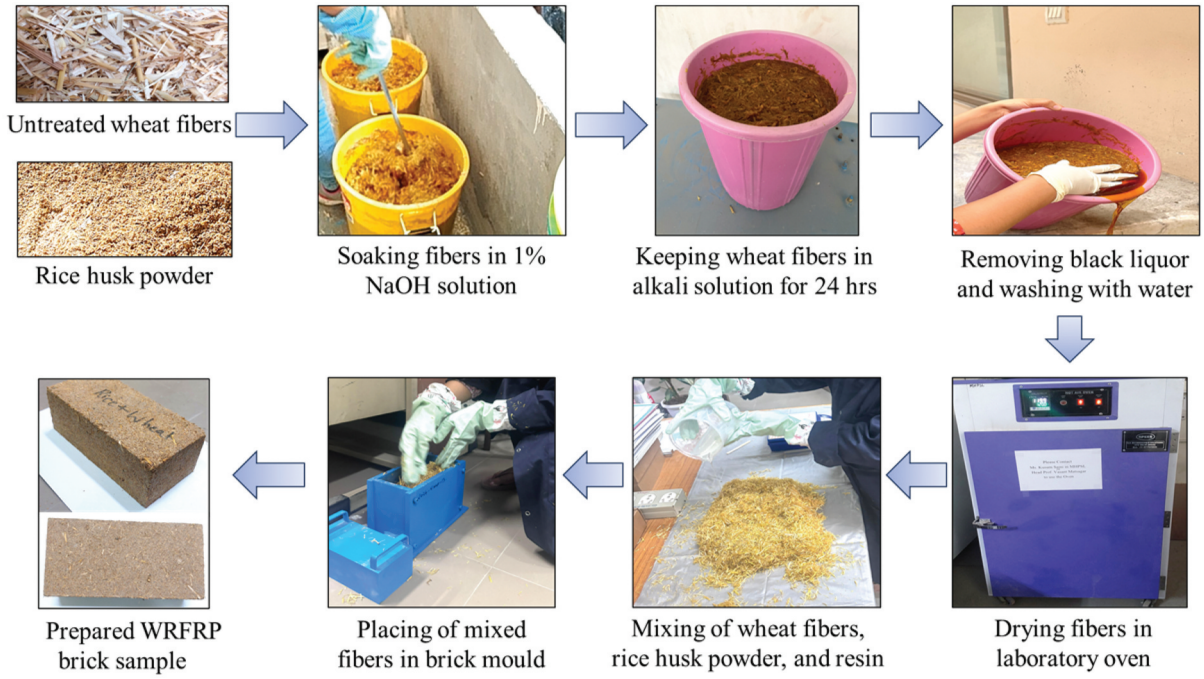


Figure 1. Preparation of wheat and rice fiber-reinforced polymer (WRFRP) brick specimens.

brick mold to eliminate trapped air bubbles, internal voids, and enhance matrix penetration between fibers (see Figure 1). The molded composite has been left to cure at room temperature (approximately 25 ± 2°C) for 24 h without disturbance, allowing sufficient time for cross-linking of the epoxy matrix. After the curing period, the composite bricks were carefully demolded and visually inspected for surface defects, uniformity, and completeness of curing prior to subsequent mechanical and thermal characterizations.

Compressive behavior of wheat and rice fiber-reinforced polymer hybrid composite at different temperatures

Wheat and rice fiber-reinforced epoxy hybrid composite specimens were exposed at room temperature, 40°C, 50°C, and 60°C before their compressive behavior was analyzed. Half-brick specimens have been tested for the compressive strength of WRFRP hybrid composites as per ASTM C67/C67M-19 (2019). Three specimens are considered for each temperature case; a total of 12 specimens are tested. Further, scanning electron microscopy (SEM) and differential scanning calorimetry (DSC) analyses have been performed to examine the effect of temperature on the microstructure of the composites.

Thermal conditioning

To evaluate the thermal response and structural stability of the fabricated biocomposite specimens at low to moderate temperatures, a systematic thermal conditioning protocol has been followed. The testing has been conducted at three distinct temperatures: 40°C, 50°C, and 60°C, selected to simulate moderate thermal exposure conditions relevant to typical environmental and service scenarios in construction and structural applications.

Specimens have been placed in a furnace equipped with precise temperature control at each target temperature, as shown in Figure 2. The furnace has been pre-calibrated to maintain a uniform thermal distribution. The specimens have been heated at a controlled ramp rate of 1°C per minute, chosen to minimize thermal gradients within the material. Upon reaching the desired temperature, specimens have been held isothermally at the target temperature for 60 min. Such conditioning enables accurate assessment of thermal softening, matrix degradation, and interfacial debonding that might occur under thermal exposure. After the conditioning period, the specimens have been removed from the furnace and allowed

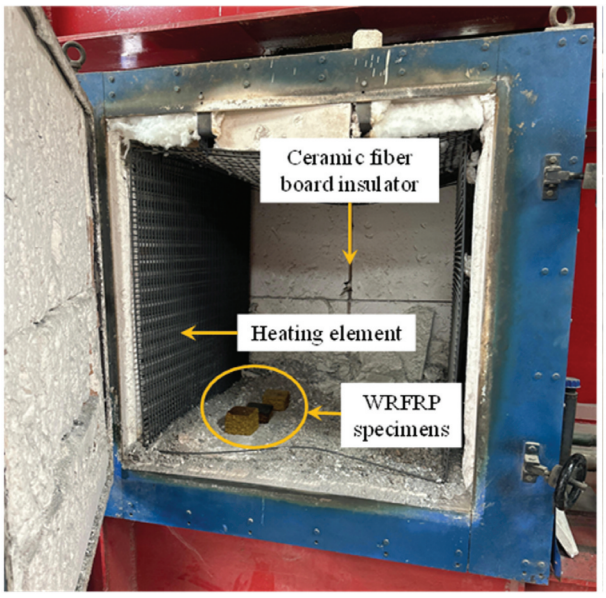


Figure 2. Thermal conditioning of wheat and rice fiber-reinforced polymer (WRFRP) hybrid composite specimens in a furnace.

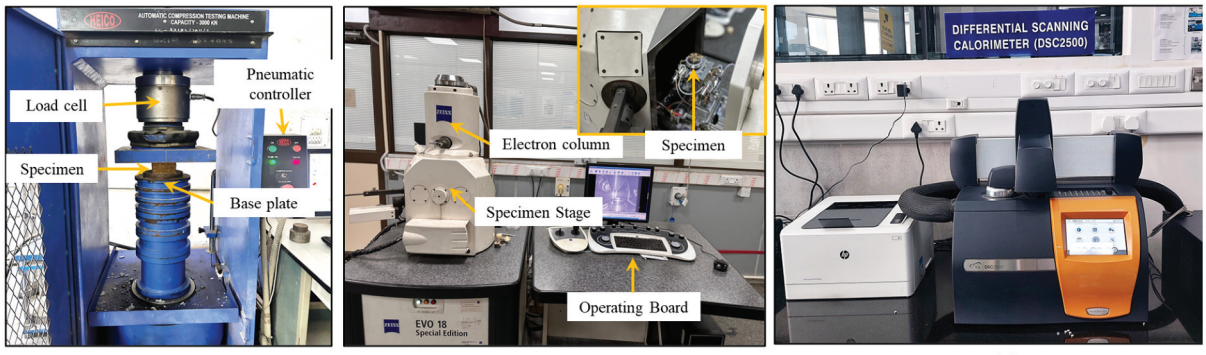


Figure 3. Test setups of (a) compression testing, (b) scanning electron microscopy (SEM) analysis, and (c) differential scanning calorimetry (DSC) analysis of wheat and rice fiber-reinforced polymer (WRFRP) composite specimens.

to cool at room temperature for 2 to 3 days before further testing. The thermally exposed specimens have been subsequently subjected to compressive strength testing and microstructural analysis, as described in the following sections.

Compressive strength testing

The compressive behavior of WRFRP hybrid composite specimens has been assessed using an automatic compression testing machine (CTM) with a load capacity of 3000 kN, as shown in Figure 3(a). The tests have been conducted in strain-controlled mode to ensure precise monitoring of material deformation under compressive loading. A crosshead speed of 0.05 mm/s has been employed, which falls within the standard quasi-static loading conditions recommended for compression tests on brittle or semi-brittle composite specimens. The slow loading rate reduces the possibility of dynamic effects or sudden failure, which is especially important for heterogeneous materials, e.g., natural fiber-reinforced composites. The testing procedure has been implemented in accordance with the testing guidelines for bricks outlined in ASTM C67/C67M-19 (2019). The collected stress–strain data provided insights into the temperature-dependent degradation of mechanical performance in

WRFRP hybrid composites. The curves are drawn based on 500 to 1000 data points; however, to avoid clutter, only a few data points are highlighted with symbols on the lines in Results and discussion.

Scanning electron microscopy (SEM) analysis

Scanning electron microscopy (SEM) analysis of WRFRP hybrid composites, thermally exposed at 40°C, 50°C, and 60°C, has been conducted, and the test setup is presented in Figure 3(b). We investigate thermal damage that might occur on the surfaces and interfaces of these composites and reveal how heat influences their structure. Specimen preparation and imaging conditions have been carefully controlled to ensure meaningful SEM observations. After exposing the composites to heat, small coupons from each specimen have been cut. To prevent charging issues with these non-conductive natural fiber composites when under the electron beam, the specimens have been mounted on aluminum stubs using carbon tape, and a thin layer of conductive gold (about 10 nm) has been applied through sputter coating (Paktin et al. 2024). This coating prevents electron buildup and yields clear high-resolution images of fracture surfaces and exposed cross-sections (Bazan et al. 2020). SEM imaging has been carried out using a Zeiss EVO 18 Special Edition SEM equipped with a LaB6 or W emitter. For the composite analysis, an accelerating voltage of 15–20 kV is typically used to achieve a balance between surface details and charge mitigation. The magnification capability (up to ~1,000,000×) and large analytical field of view (~6 mm at optimal working distance) of the microscope allowed low-magnification surveys of overall fracture topology and high-magnification inspection of microstructural details. Multiple regions of each specimen (external surfaces and internal fracture planes) have been imaged to identify key features. The SEM micrographs have been analyzed to qualitatively assess surface erosion (matrix pitting or fiber exposure), fiber–matrix interfacial bonding (evidence of fiber pull-out and fiber fracture), presence of voids/cavities, and any crack initiation or propagation paths within the matrix for natural fiber-reinforced polymer composites (Daba et al. 2024). Please note that all microstructural damages in the specimens reported are assessed after cooling down process. Therefore, all damages specified are irreversible.

Differential scanning calorimetry (DSC) analysis

The differential scanning calorimetry (DSC) analysis of WRFRP hybrid composites is performed in the study, and the test setup DSC2500 utilized by TA instruments is shown in Figure 3(c). The temperature has been varied at a rate of 5°C/min with a nitrogen gas flow rate of 20 ml/min in an inert environment over the specimen with a mass of around 9 mg. Romero-Ocaña, Delgado, and Molina (2022) also studied the detailed thermal properties of composites made of short wheat and rice straws individually reinforced in photocurable resin, where the fibers considered varied between 0.045 mm and 0.25 mm in size, using SEM and DSC analyses. However, investigations into the developed hybrid composite remain untouched.

Results and discussion

Compressive strength tests, scanning electron microscopy (SEM), and differential scanning calorimetry (DSC) analyses have been conducted for wheat and rice fiber-reinforced polymer (WRFRP) hybrid composites exposed at the temperatures of 40°C, 50°C, and 60°C. The results revealed that the hybrid composite bricks exhibit a temperature-dependent reduction in strength. The respective stress–strain behavior and damage or failure of WRFRP composites obtained are presented (limited to 0.4 mm/mm strain) in Figures 4–8. The results indicate that even low to moderate thermal exposure has a pronounced influence on both the compressive behavior and microstructure of these WRFRP composites. Exposure to moderate temperatures led to reduced compressive strength and observable microstructural degradation, reflecting a deterioration of fiber–matrix interfacial integrity. This overview highlights the temperature sensitivity of the composites.

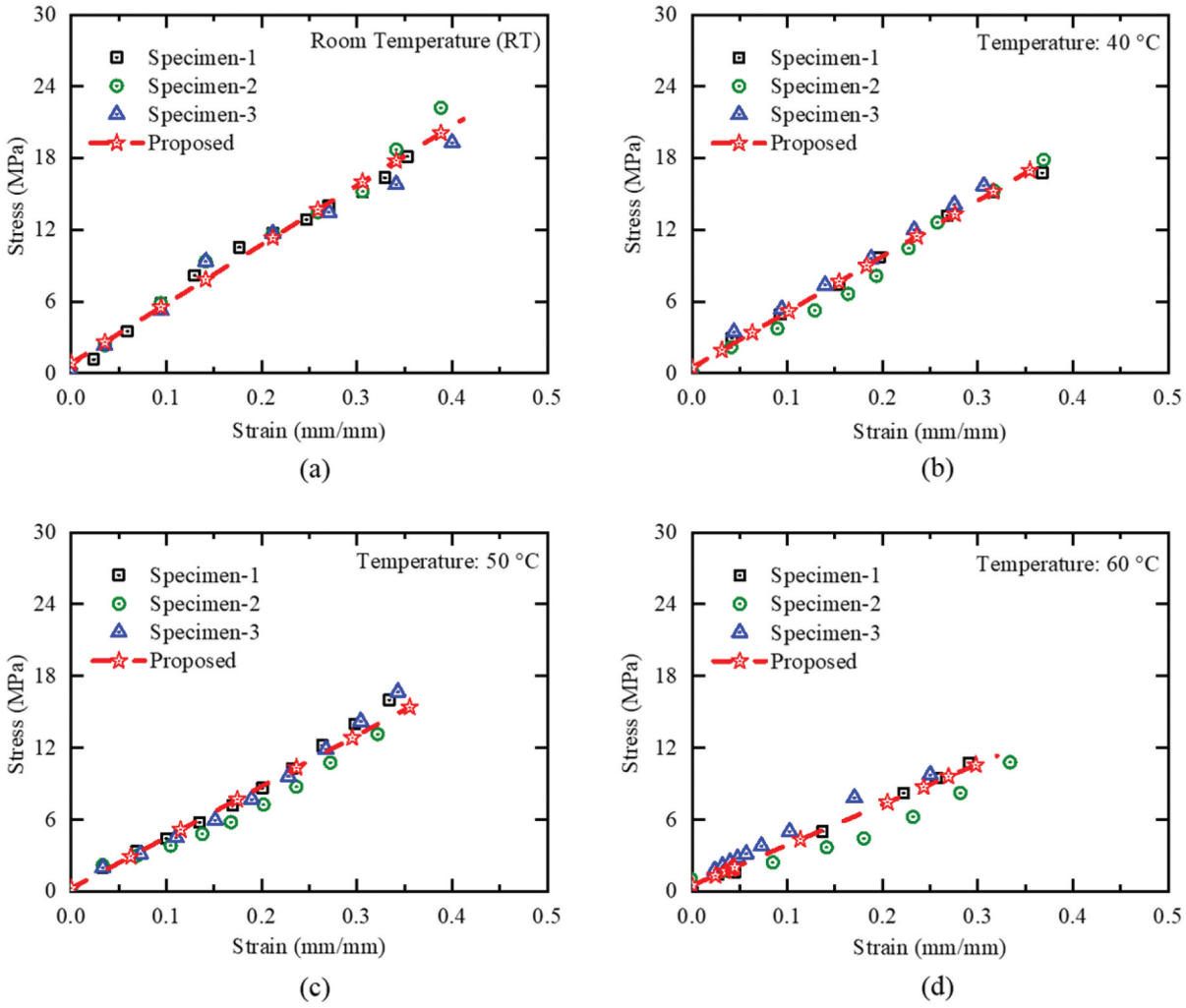


Figure 4. Stress–strain curves of wheat and rice fiber-reinforced polymer (WRFRP) exposed at (a) room temperature (RT), (b) 40°C, (c) 50°C, and (d) 60°C under compression.

Compressive behavior at different temperatures

The compressive behavior of WRFRP hybrid composites at low to moderate temperatures shows apparent thermal sensitivity and progressive degradation in mechanical strength. The average strength of the composites has been estimated at each temperature, considering a coefficient of variance (CoV) less than 20% for adequate accuracy (see Table 1). The composite exhibits its maximum compressive performance at room temperature (RT), with an average compressive strength of approximately 21.5 MPa (see Table 1). The corresponding stress–strain curves are presented in Figure 4. Bolcu, Stănescu, and Mirițoiu (2022) reported the compressive strength of around 28 MPa for a 21 mm-sized wheat fiber-reinforced epoxy composite. However, approximately 42% of the wheat fiber mass was used in the composite. The study reported a reduction in the strength and enhanced ductility due to hybridization with dammar resin.

The specimens initially exhibit a uniform linear increase in stress with strain under ambient conditions. Please note that the nonlinear behavior around breakage was not reported in this study. When the composite is exposed to the temperature of 40°C, a moderate decline in peak stress is observed, with the average compressive strength values dropping to around 18 MPa, reflecting a 16.2% reduction from that at RT. While the general trend in the stress–strain curve remains smooth and continuous, minor undulations or deviations begin to appear. These slight non-linearities are early indicators of matrix softening and suggest the possibility of microcrack initiation, particularly at fiber–matrix interfaces. However, despite this

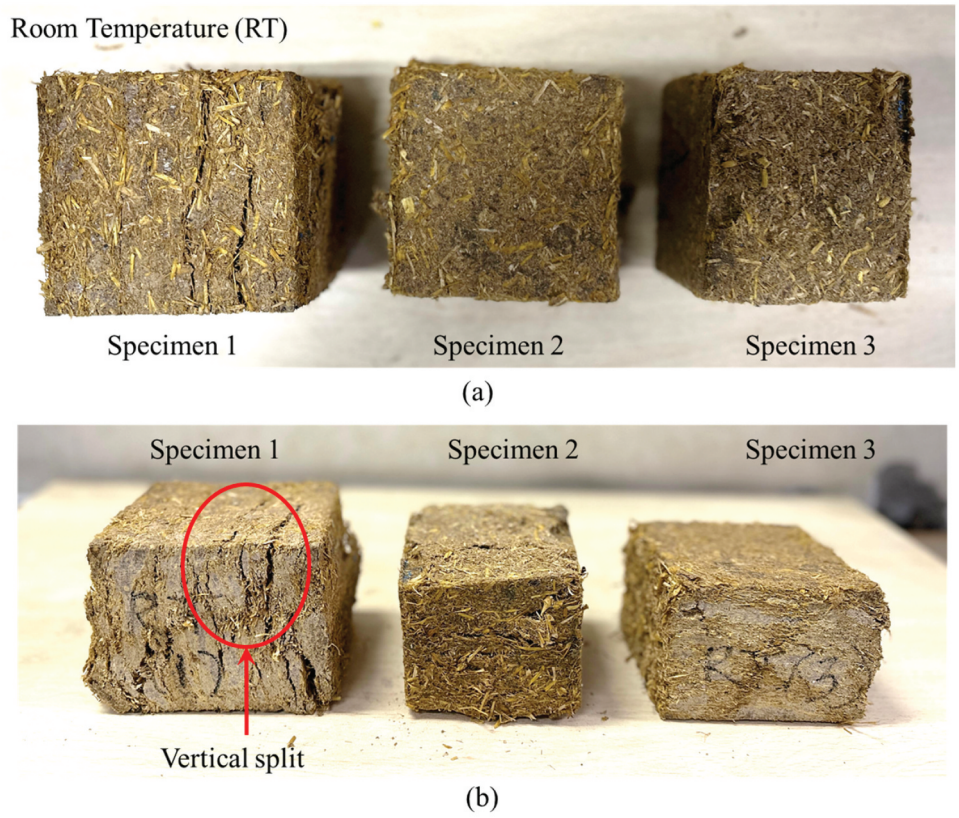


Figure 5. Failure of wheat and rice fiber-reinforced polymer (WRFRP) specimens at room temperature (RT), (a) top view and (b) front view.

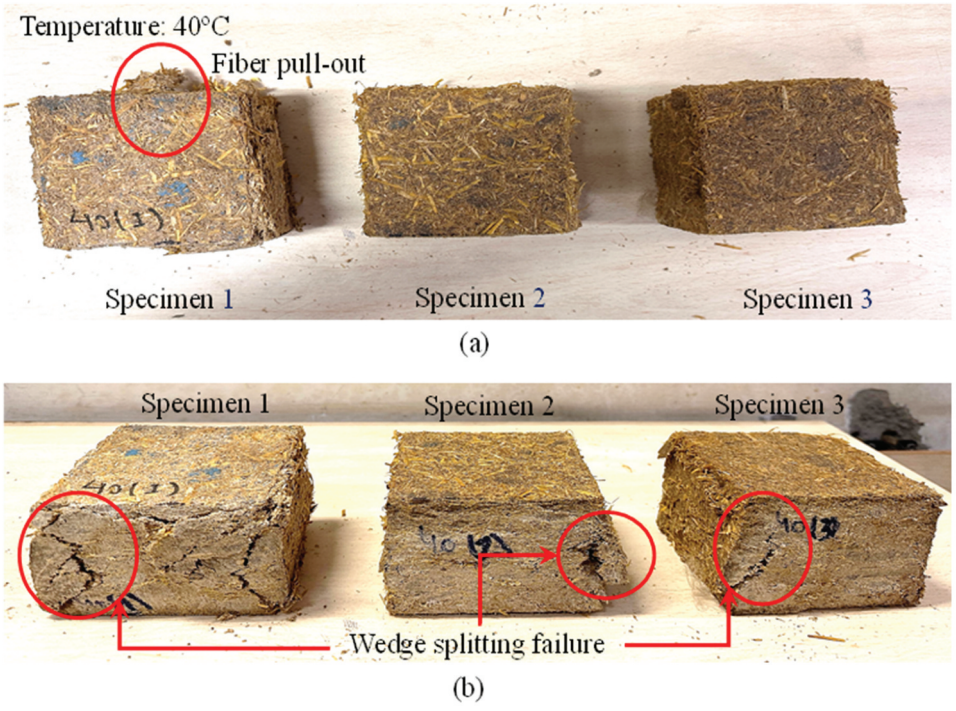


Figure 6. Failure of wheat and rice fiber-reinforced polymer (WRFRP) specimens at 40°C, (a) top view and (b) front view.

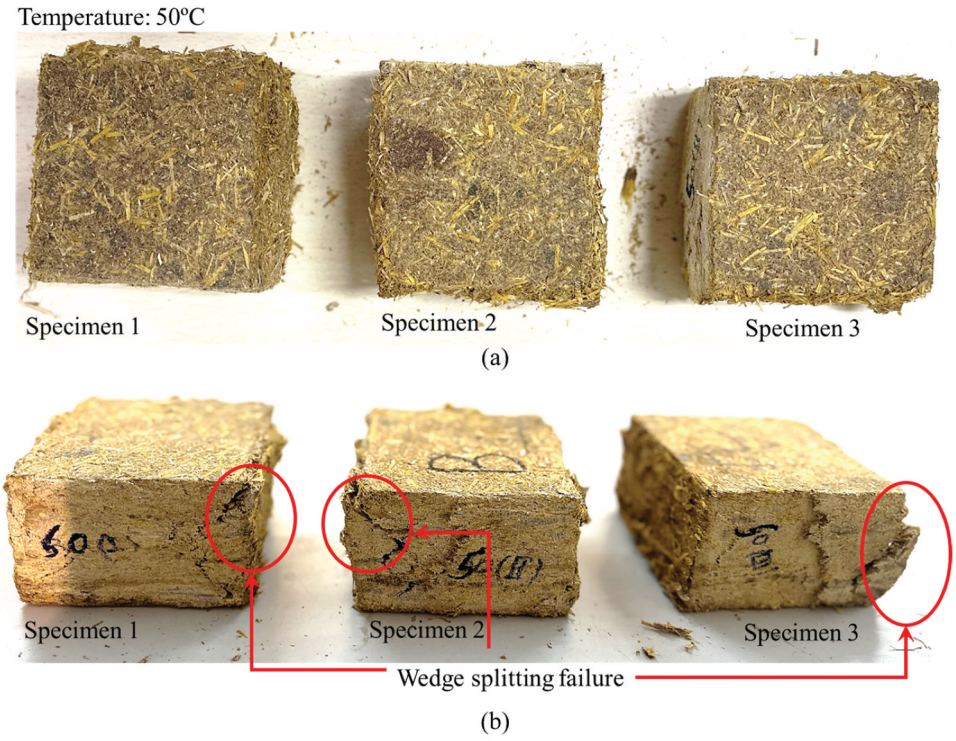


Figure 7. Failure of wheat and rice fiber-reinforced polymer (WRFRP) specimens at 50°C, (a) top view and (b) front view.

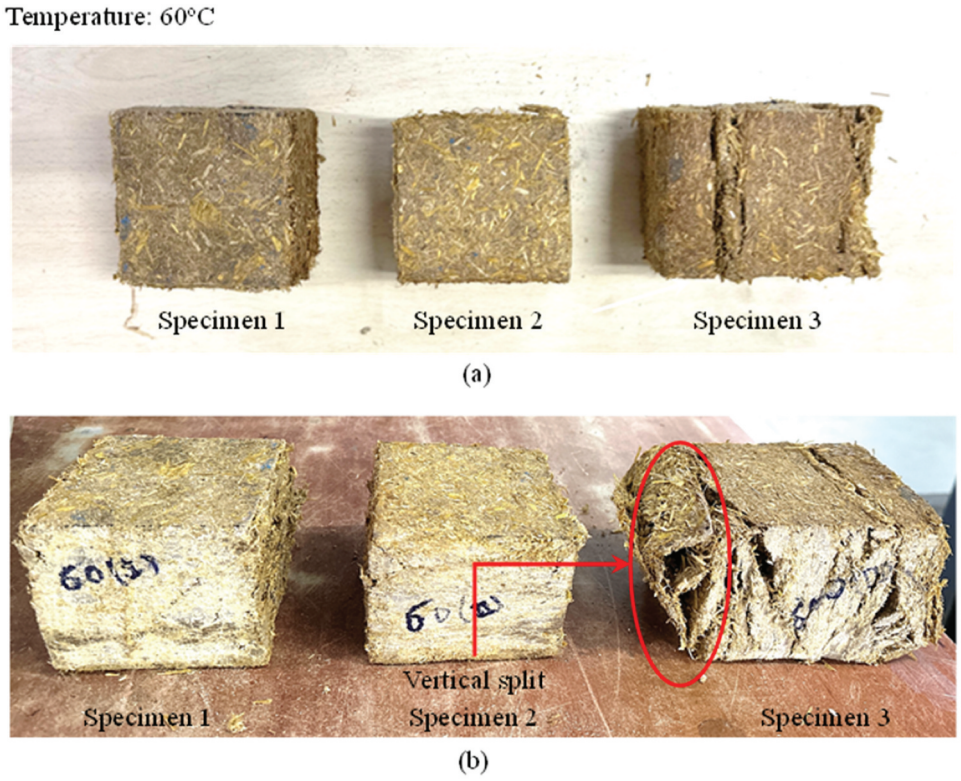


Figure 8. Failure of wheat and rice fiber-reinforced polymer (WRFRP) specimens at a temperature of 60°C, (a) top view and (b) front view.

Table 1. Compressive strength of wheat and rice fiber-reinforced polymer (WRFRP) hybrid composites at different temperatures.

Temperature	Specimen	Peak compressive strength (MPa)	Average peak compressive strength (σ_f) (MPa)	COV (%)
Room Temperature (RT)	Specimen 1	18.13	21.5	16.5
	Specimen 2	25.15		
	Specimen 3	21.05		
40°C	Specimen 1	17.60	18.0	4.5
	Specimen 2	18.88		
	Specimen 3	17.40		
50°C	Specimen 1	17.55	16.4	13.9
	Specimen 2	13.74		
	Specimen 3	17.81		
60°C	Specimen 1	11.74	10.8	9.1
	Specimen 2	10.81		
	Specimen 3	9.76		

Here, σ_f is the peak compressive strength at temperature T and the coefficient of variance, COV (%).

marginal loss in strength, the composite exhibits adequate ductility and structural consistency, suggesting it may remain viable for applications subjected to moderate thermal exposure.

At 50°C of exposure temperature, a significant decrease in the compressive strength of WRFRP composites is evident. The average compressive strength drops to approximately 16.4 MPa by approximately 24% (23.6%) from that at RT. Stress–strain curves at this temperature exhibit reduced linearity, with visible stress drops and dips, indicating localized failures such as interfacial debonding, partial debonding, and microstructural cracking. The increased presence of local instabilities in the stress–strain response further supports the notion that the composite material begins to experience thermal weakening and breakdown of the epoxy matrix at this stage.

The most severe deterioration in the strength of WRFRP hybrid composites occurred at 60°C. The compressive strength reduces drastically to an average of 10.8 MPa, indicating approximately 50% (49.8%) reduction from that of at RT. The stress–strain curves at this temperature exhibit abrupt stress declines and early failure initiation under compression. This behavior is symptomatic of advanced matrix decomposition, where the polymer network loses its stiffness and fails to resist compressive loads. The material is likely nearing or exceeding its glass transition temperature (T_g); at this point, the epoxy matrix undergoes significant softening, resulting in a loss of rigidity, and mechanical stability. Failure occurs at lower strain levels, with reduced load-bearing capacity and loss of ductility.

Under compressive loading across all tested temperatures, the WRFRP specimens displayed distinct failure modes that were primarily categorized as wedge-split failure and vertical splitting. Wedge-split failure, observed in specimens at 40°C and 50°C (refer to Figures 6 and 7), is characterized by the development of cracks along two inclined shear planes, which propagate inward to form a wedge-shaped fracture. This mode of failure often arises from debonding or tensile cracking and is a common consequence of stress concentrations at the specimen ends or internal flaws (Opelt, Cândido, and Rezende 2018). On the other hand, vertical splitting involves cracks propagating in the direction parallel to the applied load, typically along the longitudinal axis of the specimen. This failure is frequently observed when lateral confinement is limited, allowing axial cracks to form and extend readily. The vertical splitting, observed in specimens at RT and 60°C (see Figures 5 and 8), suggests that the fibers, while contributing to initial load resistance, lose effectiveness in later stages due to interfacial debonding and fiber pull-out, especially as temperature increases.

Based on the compressive stress–strain behavior at low to moderate temperatures, the following equations are proposed to estimate compressive strength at intermediate temperatures. Please note that the proposed equation does not specify or highlight the nonlinearity created in the behavior around the breakage point. The stress–strain relations at any temperature, T, are represented in Equation 1. The constant parameters of the equation can be calculated based on the temperature rise with respect to room temperature using Equation 2 and Equation 3. Here, $\bar{\varepsilon}$ is strain, and $\bar{\sigma}_f$ represents stress at temperature, T, presented as follows,

$$\bar{\sigma}_f = A\bar{\varepsilon} + B. \tag{1}$$

The temperature-dependent constants in Equation 1 are obtained as,

$$A = A_0(1 + 1.1 \times 10^{-3}\Delta T - 30 \times 10^{-5}\Delta T^2), \tag{2}$$

$$B = B_0(1 - 0.071 \times 10^{-1}\Delta T - 2.5 \times 10^{-3}\Delta T^2 + 7.0 \times 10^{-5}\Delta T^3). \tag{3}$$

Based on the ratio of peak compressive strength, σ_f at temperature, T, and peak compressive strength, σ_0 at RT, A_0 and B_0 are elastic constants at RT, with respect to the increment in temperature (ΔT), the following relation is proposed for temperature-dependent stress with a coefficient of determination, $r^2 > 0.99$,

$$\sigma_f = \sigma_0(1 - 0.24 \times 10^{-1}\Delta T + 1.4 \times 10^{-3}\Delta T^2 - 3.0 \times 10^{-5}\Delta T^3). \tag{4}$$

Microstructural degradation and thermal analysis of WRFRP composite

SEM images from the SEM analysis reveal microstructural degradation in WRFRP hybrid composites with increasing thermal exposure from RT to 60°C (c.f., Figure 9). Here, clean fiber surfaces show strong interfacial bonding in the composite at RT with low porosity or cavities compared to other temperatures (refer to Figure 9(a)). At RT, the fiber–matrix interface appears well bonded; however, at 40°C, early interfacial debonding is observed as a gap between the fiber and the matrix (Gairola et al. 2024). At 50°C, thermal stress and weakening of the epoxy matrix cause more noticeable damage, with matrix microcracks forming around the fibers and the interface deteriorating (Mokhothu and John 2017). Fibers begin to pull out from the epoxy, leaving voids on fracture surfaces – clear evidence that the fiber–matrix adhesion has weakened. This aligns with the epoxy approaching its glass transition temperature (~60°C), leading to a decrease in rigidity (Oun et al. 2022). Many fibers are entirely debonded and projecting from the matrix, and the epoxy displays plastic deformation and cracking (Mokhothu and John 2017).

At 60°C, the composite shows widespread fiber pull-out, fiber fracture, and interfacial failure. Furthermore, natural constituents of the wheat fiber, such as residual starch, become thermally active at high temperatures and in the presence of moisture, and thus starch can gelatinize (swell and lose its crystalline structure) (Zhuang et al. 2024), contributing to fiber weakening and interface breakdown. The composite gradually deteriorates with increasing temperature; the bond between fiber and matrix degrades through debonding and fiber pull-out, while the epoxy matrix softens and splits, leading to a compounding loss of microscale structural integrity. However, early indicators of interfacial weakening start to show at 40°C, including partial debonding and roughened matrix surfaces, suggesting the start of matrix softening and weakening fiber–matrix adhesion.

Also, some detached resin particles and fiber pull-out were observed (refer to Figure 9(b)). The degradation became more severe by 50°C; the matrix displays deeper cracking and resin fragmentation, and fibers showed partial debonding. Reduced epoxy stiffness, coupled with the influence of temperature-induced expansion and weakening of natural fiber ingredients (such as hemicellulose), speeds up failure processes (Mokhothu and John 2017), see Figure 9(c). While the epoxy was observed as plastically deformed and porous at 60°C, the SEM micrographs show catastrophic microstructural collapse of fibers from the matrix, pull out, or split. Zhuang et al. (2024) also observed similar patterns where the matrix approaches its glass transition temperature and the fibers experience biochemical transitions, including starch gelatinization and cellulose breakdown. Low to moderate temperature exposure damages the structural integrity of the WRFRP hybrid composites, resulting from numerous factors including interfacial debonding, fiber degradation, and resin softening; therefore, underlining the thermal limits of such biocomposites (Oun et al. 2022). These microstructural damages due to the mentioned temperature exposure affect the overall mechanical properties, e.g., reduction in compressive strength of the composites.

It has been observed that the endothermic reaction started at about 15°C, reaching a peak around 64°C, moisture evaporation (Te), representing the beginning of evaporation and heat deflection in resin. The glass transition (T_g) occurs between 80°C and 170°C, peaking at 89°C, in WRFRP hybrid composites, see Figure 10. Around this temperature range, rapid moisture loss followed by the loss of lignin in fibers takes place. The alkali treatment helps in reducing water absorption by the fibers, resulting in higher temperature points as compared to untreated fiber-

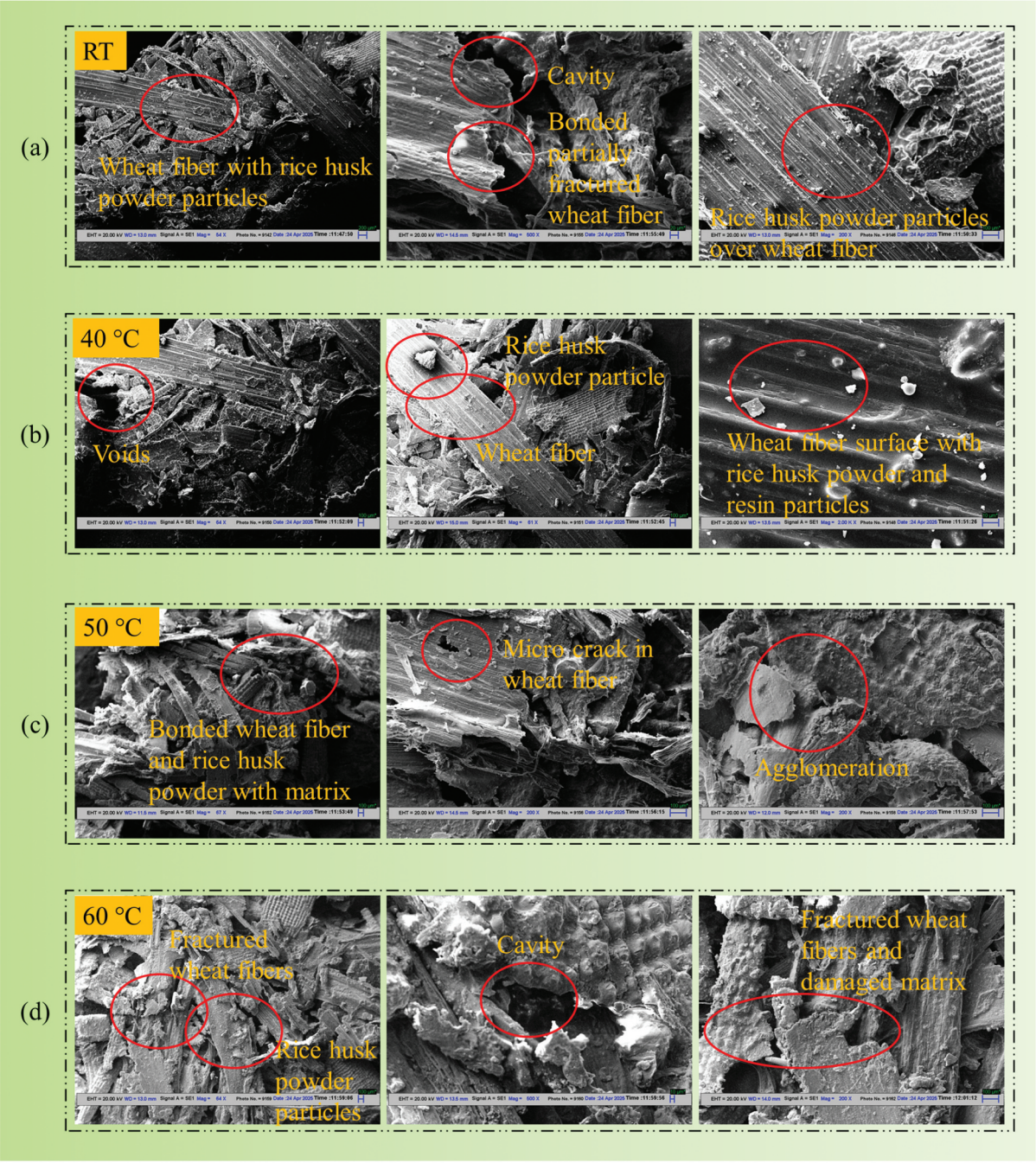


Figure 9. Microstructural degradation of wheat and rice fiber-reinforced polymer (WRFRP) composites under thermal exposure at (a) room temperature (RT), (b) 40°C, (c) 50°C, and (d) 60°C.

based composite (Haque et al. 2021). The endothermic reaction starts reaching peak, showing polymerization of unreacted monomers around 171°C, followed by a peak at 236°C corresponding to the fusion temperature in the composite. In this temperature range, the hemicellulose and cellulose in fibers begin to decompose. Beyond 280°C, the exothermic reaction, followed by the endothermic reaction, represents the initiation of total loss of lignin of the fibers and cross-linking, respectively. No exothermic reaction up to this temperature represents thermal stability in the composite before the melting point arrives (Madival et al. 2023). Similar thermal patterns were observed for the composites made of 5% wt wheat straw and 5% wt rice straw, individually reinforced in a photocurable resin, with slight variations in the temperature points (Romero-Ocaña, Delgado, and Molina 2022).

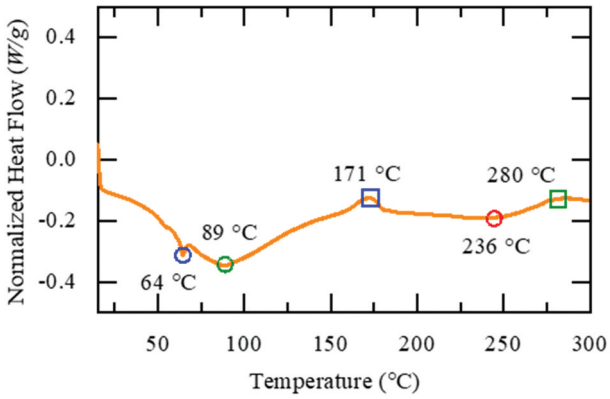


Figure 10. Differential scanning calorimetry (DSC) analysis of WRFRP hybrid composites, normalized heat flow over the specimen with temperature.

Conclusions

In this study, the compressive and microstructural degradation behavior of the novel wheat and rice fiber-reinforced polymer (WRFRP) hybrid composite bricks is investigated under thermal exposure at room temperature, 40°C, 50°C, and 60°C. The fabrication process for the developed composites, using hand lay-up and compression techniques, is also presented in the study. In addition, scanning electron microscopy (SEM) and differential scanning calorimetry (DSC) were conducted to examine microstructural changes in fiber–matrix bonding, degradation patterns, and thermal behavior at low to moderate temperatures. Accordingly, the following conclusions are drawn from the study.

(1) Wheat and rice fiber-reinforced polymer (WRFRP) hybrid composite showed an average compressive strength of 21.5 MPa at room temperature. The strength gradually decreases to about 24% till 50°C, and then the reduction is drastic, reaching 50% at 60°C.
(2) The stress–strain response of WRFRP composite is approximately linear under compressive loading till failure occurs in brittle mode. The variation in the peak compressive strength of WRFRP at temperature, T, with respect to increment in the temperature can be represented via a cubic relation.
(3) Vertical splitting, wedge splitting, and fiber pull-out are major failure modes in WRFRP composite bricks when exposed at room temperature, 40°C, 50°C, and 60°C under compressive loading.
(4) The WRFRP composites show microstructural stability up to 50°C with better particle packing and interface quality. At 60°C, adverse degradation occurs in the form of matrix cracking, fiber debonding, and fiber damage.
(5) In WRFRP composites, the glass transition (T_g) occurs between 80.0°C and 170.0°C with a peak at 89°C.

The performance of such biocomposites is dependent on various parameters, inherent properties based on the environment at the origin of agricultural residues, processing or treatment of the fibers, fabrication process of the composite, composition, exposed hygrothermal environment and its duration before and during testing, and testing loading rate. The effects of the mentioned parameters on agricultural-residue-based biocomposites can be studied in the future. The results presented in this study can be used to approximate the compression behavior and thermal response of the developed composites. Accordingly, the use and design of the developed natural fiber-reinforced composites/biocomposites can be selected for the desired applications.

Author contributions

CRediT: **Umakant Pandey:** Data curation, Formal analysis, Investigation, Methodology, Validation, Writing – original draft, Writing – review & editing; **Kusum Saini:** Conceptualization, Formal analysis, Funding acquisition, Investigation, Methodology, Supervision, Validation, Writing – original draft, Writing – review & editing; **Vasant A. Matsagar:** Conceptualization, Formal analysis, Funding acquisition, Investigation, Methodology, Project administration, Resources, Supervision, Validation, Writing – review & editing; **Venkatesh Kodur:** Conceptualization, Funding acquisition, Investigation, Methodology, Resources, Supervision, Validation, Writing – review & editing.

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