RSC Advances

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Cite this: *RSC Adv.*, 2025, **15**, 9534

Mechanically robust and thermally insulating natural cotton fiber-reinforced biocomposite panels for structural applications†

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Natural cotton fiber-reinforced heat-insulating biocomposites with high mechanical strength were designed and developed in the present research for various structural applications. Novel cotton-reinforced polypropylene (PP) composites with improved interfacial adhesion were fabricated through uniform blending of cotton and PP fibers of different volume fractions for high mechanical strength. The developed cotton–PP specimens were characterized by examining their tensile, flexural and impact strength, heat barrier properties, and thermal stability at high temperatures. Among the composites, the material with a cotton and PP ratio of 40/60 demonstrated a maximum tensile and flexural strength of 62.09 MPa and 138.90 MPa, respectively, whereas the 50/50 cotton composite showed a maximum impact strength of 130.75 kJ m^{-2}. A decrease in tensile and flexural strength was noticed with increasing the reinforced cotton fiber in composites. In the case of thermal performance, however, specimen 60/40 showed the lowest thermal conductivity (0.063 W m^{-1} K^{-1}) and the highest conductive heat resistance (0.063 $m^2 \cdot K$ W^{-1}). The composite 60/40, after exposure to radiant heat, also exhibited a maximum radiant heat resistance with the lowest surface temperature of 32.0 °C. Thermogravimetric analysis and differential scanning calorimetry showed adequate thermal stability and heat energy-absorbing capability of materials at elevated temperatures. The outcomes of the present study revealed that cotton–PP composites developed through uniform blending of fibers possess superior mechanical strength and adequate thermal insulation properties and suggested the practicability of using them in various structures where mechanical and thermal performance are the key requirements.

Received 9th January 2025
Accepted 21st March 2025

DOI: 10.1039/d5ra00213c

rsc.li/rsc-advances

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1 Introduction

Natural fiber-reinforced composites (NFRCs) with high strength have gained significant attention from researchers in recent years as lightweight engineered materials for specific applications.[1] In addition to high mechanical strength, NFRCs also exhibit several unique characteristics, for example, excellent mechanical performance, sufficient thermal insulation, non-abrasiveness to equipment, and resistance to corrosion and wear.[2,3] Such exceptional properties have led NFRCs to find utilization in diverse fields, such as aircraft, railways, automobiles, marine, and so on.[4,5] Because of their suitable mechanical strength and low heat conductivity, several contemporary research studies showed the potential application of such types of lightweight and sustainable composites for thermal insulation in building engineering.[6,7] In order to fabricate NFRCs, cellulosic fibers, being lower cost, nontoxic, naturally available, renewable, and having low heat conductivity, are commonly used as the reinforcement material,[8,9] whereas synthetic polymers, both thermosets and thermoplastics, are utilized as the matrix material to support the fibers and possesses distinct advantages and drawbacks.[10,11] However, thermoplastic polymeric materials offer several notable benefits, for instance, convenient processing, recyclability, low cost, and manufacturing simplicity over thermosetters that are widely utilized as the matrix materials in NFRCs.[12]

Thermoplastics, such as polyethylene, polypropylene, and polyvinyl chloride, are the prevalent matrix materials that are commonly used with natural fiber in fabricating fiber-reinforced composites.[13] Among them polypropylene (PP), having the advantage of better strength, stiffness, chemical resistance, and thermal insulation, has established itself as a potential matrix polymer for utilization in NFRCs.[14,15] However, the incompatibility between cellulosic fiber and PP arises from their contrasting hydrophilic and hydrophobic nature, leading to weak bonding at their interface.[16] This improper attachment creates a scope of forming voids inside

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† Electronic supplementary information (ESI) available. See DOI: https://doi.org/10.1039/d5ra00213c

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the material during the fabrication leading to diminished progression of applied stress between the matrix and the reinforcement, resulting in the production of a composite with inferior physical properties associated with mechanical strength and durability.[17] The interfacial characteristics of the reinforced fiber and the matrix are therefore considered a significant issue for assessing the mechanical properties, while insufficient interfacial adhesion between the constituents eventually results in an inferior strength and stiffness of the composite materials.

To overcome the shortcomings of NFRCs regarding mechanical characteristics, currently, various physical and chemical treatments for natural fibers are available for improving the interfacial interaction and compatibility between reinforced fiber and matrix.[18] Physical techniques, such as needle punching, were employed to enhance the interaction between reinforced natural fiber and thermoset matrix, consequently, improving the mechanical strength of composites.[19,20] Chemical modifications for enhancing the adhesion of natural fibers with hydrophobic polymer matrix have been investigated in several contemporary studies. By employing chemical treatment, the interface of natural fibers is optimized by introducing different moieties or stimulating hydroxyl (–OH) groups that can efficiently attach with the polymer matrix.[21] One of the commonly used chemical methods for treating natural cellulosic fibers to reinforce thermosets and thermoplastics is known as alkali treatment or mercerization. Baccouch and coworkers showed a maximum increase in 70.0% Young's modulus of cotton fiber reinforced composites treated with caustic soda (NaOH).[22] Li and coworkers reported improved tensile strength, tensile modulus, and thermal stability of composites after consecutive treatments of cotton fiber with alkali and copper ethanolamine solutions.[23] Bodur and coworkers investigated the effects of silane, alkali–silane, maleic anhydride, and alkali–maleic anhydride coupling agent treatment of textile fiber and demonstrated 60–70% improvement of composite's strength *via* enhancing compatibility between fiber and matrix.[24]

The chemical treatment of fibers using different techniques, however, is time-consuming and complicated, with some negative impacts on the physical properties of fibers.[21] In this regard, enhancing interfacial bonding by modifying the physical interaction between reinforced fiber and the matrix is advantageous where no extra processing time and chemicals are required. Nevertheless, current scientific literature lacks substantial studies concerning the enhancement of interaction through physical modification using different techniques of composite preparation. Among the techniques for fabricating composites, compression molding is considered a method, which offers several benefits, including low cost, minimum waste, and compatibility for manufacturing large and complex parts.[25] Developing fiber-reinforced composites using the compression molding technique typically involves vertical heating and pressing of materials.[16] However, due to vertical pressing, thermoplastics, after melting, flow unidirectionally, which results in inadequate impregnation of fibers and limits a robust and uniform bonding throughout the composite between reinforcement and matrix when they are stacked in layers in the metal die of the machine. In this regard, a bidirectional flow of melted resins can be considered a potential approach for proper impregnation of fiber into the matrix, which would eventually enhance the physical interaction between them.

A homogeneous mixing of reinforcement and matrix in the form of fiber is therefore employed as a composite preform in the present study for enhancing the interaction at the interface of composites. Because of intimate blending and the distribution of thermoplastic matrix around the cotton fibers in the composite preform uniformly, the melted PP resins during compression molding are expected to flow bidirectionally and develop a consistent bonding around the reinforced fiber after the heat press, which results in improved mechanical performance of materials. Fabricating NFRCs with superior mechanical strength, however, has a negative impact on the thermal insulation properties because of improved interfacial adhesion and reduced voids in the materials.[26] Therefore, designing NFRCs with better mechanical properties and sufficient thermal resistance is challenging and crucial in optimizing both characteristics in the same material intended for structural applications.

Hence, the fabrication of high mechanical strength and thermal insulating NFRCs by uniform blending of reinforcement and matrix fibers for improved interfacial adhesion has been proposed in this work. The present study aimed to develop cotton-reinforced polymeric composites using different volume fractions of natural cotton and PP fibers followed by the investigation of their mechanical and thermal characteristics, including tensile and flexural strength, thermal conductivity, resistance to conductive and radiative heat, differential scanning calorimetry, and thermogravimetric analysis for their potential applications in various engineering fields. It also aimed to fabricate composites with optimized reinforced fiber and matrix for enhanced heat barrier properties to assess the practicality of using them in various structures where both mechanical and thermal performances are major concerns.

2 Experimental

2.1 Materials

The composite, in the current research, was fabricated by utilizing cotton and PP staple fibers as reinforcement and matrix, respectively. The cotton fiber was collected from the local market and used as received. PP fiber was procured from Tianjin Greentech Chemical Fiber Co., Ltd, China. The physical characteristics of cotton and PP fibers are listed in Table 1.

2.2 Methods

2.2.1 Composite preparation. The uniform blending of reinforced cotton and PP fibers for developing composites involved an initial opening and hand mixing of fibers with required volume fractions to prepare mixed lap. The hand mixing was carried out to ensure individualization and mixing of cotton and PP fibers appropriately. The hand-mixed fibers

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Table 1 Physical parameters and relevant values of cotton and polypropylene fiber

Parameter	Cotton fiber	Polypropylene fiber
Fiber type	Staple fiber	Staple fiber
Length	22–34 mm	38 mm
Density	1.54 g cm^{-3}	0.92 g cm^{-3}
Color	Off white	White
Grade	100% virgin	100% virgin
Linear density	1.3 denier	1.5 denier

were then fed into the feeding zone of the miniature blowroom (Digital Blowing Machine, DSBL, China) and delivered as fiber flocks after opening, cleaning, and blending precisely. The blended fiber flocks were then passed into a carding machine (Digital Carding Machine, DSBL, China) to prepare a thin lap by uniform parallelization and individualization of fibers (Fig. 1). The prepared carded lap with the required thickness was collected from the delivery roller of the machine and dried properly at 60 °C for 30 minutes using an oven dryer to eliminate the residual moisture from the fiber. An elevated temperature during the drying was avoided to minimize the degradation of fiber quality, especially the thermoplastic PP fibers that are sensitive to heat.

The dried carded lap was cut (170 mm × 170 mm) using scissors and placed in layers into the metallic frame (170 mm × 170 mm × 4 mm) of the machine. The number of layers for each composite was determined based on the calculated weight of the specific cotton–PP ratio of the material. Then, the composite was prepared by heat pressing the preform in layers at 190 °C (above the melting point of PP) using a compression molding machine (38914NE100, CARVER, USA). The blended preform, during the fabrication of composites, was pressed with a 17.0 kg cm^{-2} pressure for 15 minutes to avoid any deterioration of fiber quality. Four different volume fractions of cotton and PP fiber, such as 30 : 70, 40 : 60, 50 : 50, and 60 : 40, were used to develop composites for experimental investigation. The information about the developed composites of different volume fractions and coding for ease of identification is given in Table 2.

2.2.2 Material characterization

2.2.2.1 Mechanical performance. The tensile strength test of composites was performed using the universal testing machine (AG-X Plus, Shimadzu, Japan) according to the ASTM-D638 standard. Test specimens were cut into 165 mm × 13 mm × 4 mm, and a standard gauge length of 50 mm between two jaws with a strain rate of 5 mm min^{-1} was maintained during the experiment.

The flexural strength of composites was determined using the same apparatus employed in the tensile experiment following ASTM D790 standard with a three-point bending technique. During the experiment, the specimens (127 mm × 12.7 mm × 4 mm) were strained at a rate of 1.40 mm min^{-1}, keeping the distance between the spans was 64 mm.

The impact strength testing was conducted using the Charpy impact test apparatus following the ASTM D6110-18. An unnotched sample with the dimension of 127 mm × 10 mm × 4 mm was used for measuring the impact strength, and the corrected energy absorbed during the test was recorded. The impact strength of the developed composites was calculated using eqn (1).

$$\text{Impact strength} = \frac{\text{impact energy}}{\text{area}} \text{ kJ m}^{-2} \quad (1)$$

The test specimens were cut using a Bosch angle cutter fitted with a 125 mm carbide multi-wheel cutting disc. During the cutting of composites, accuracy and the disc's sharpness guaranteed no material's deformation during the sample preparation. Five specimens for each test composite were used for

Fig. 1 Schematic diagram of developing cotton–PP composite through uniform blending of reinforced cotton and PP fibers.

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Table 2 The naming of the composite specimens with density and thickness (mean ± SD) as measured

Fiber component	Material coding	Density (kg m^{-3})	Thickness (mm)
Cotton–PP (30 : 70)	CPP1	1096 ± 1.96	4.09 ± 0.04
Cotton–PP (40 : 60)	CPP2	1103 ± 2.13	4.14 ± 0.05
Cotton–PP (50 : 50)	CPP3	1113 ± 1.98	4.17 ± 0.04
Cotton–PP (60 : 40)	CPP4	1118 ± 2.26	4.19 ± 0.06

tensile, flexural and impact strength analysis, and the mean value was presented.

2.2.2.2 Morphological analysis. The morphological examination was conducted using a scanning electron microscope (SEM) (Phenom Pro G6, Netherland). A low vacuum mode with an accelerating voltage of 10 kV was used during the investigation. Before the test, specimen surfaces were gold (Au) coated using a coating machine (Model Q 150 R from Quorum, England).

2.2.2.3 Thermal barrier performance. The thermal conductivity of test composite specimens was assessed using Lee's disc method. The investigation was carried out using the device with two uniform copper discs, a precise heat source (guarded hotplate) and two heat sensors. A constant temperature (60 °C) of the guarded hotplate was maintained during the experiment. Thermal conductivity and resistance are calculated using the following eqn (2)–(4).

$$\text{Thermal conductivity, } K = \frac{Q \times d}{A(T_1 - T_2)} \quad (2)$$

$$\text{Heat flow rate, } Q = mc\frac{\mathrm{d}T}{\mathrm{d}t} \quad (3)$$

$$\text{Thermal resistance, } R = \frac{d}{K} \quad (4)$$

K = thermal conductivity (W m^{-1} K^{-1}), A = sample area (m^2), T_1 and T_2 = temperatures in kelvin, d = thickness of sample (m), Q = rate of heat flow (W), m = mass of the specimen (kg), c = specific heat (J kg^{-1} K^{-1}), $\frac{\mathrm{d}T}{\mathrm{d}t}$ = cooling rate (K s^{-1}).

The test specimens' resistance to radiative heat was evaluated using a bench-scale test apparatus. After subjecting the composite materials to the radiance of a 100 W incandescent lamp positioned 20.0 cm away, the resistance of the composites to radiant heat was measured. The temperature of the side exposed to radiant heat of the composite and the temperature of the composite exposed to the environment (25.0 ± 0.1 °C and 65 ± 2% RH) were documented every 10 seconds over a 60 minute duration using two thermocouples.

The FLIR T400-series thermographic infrared camera was utilized to observe changes in the surface temperature of a material placed on a hot surface at 50.0 ± 0.1 °C. Subsequently, images were captured over a period of 120 s from 1000 mm above the material's surface. Each specimen's maximum and minimum temperatures were recorded during this period. Standard atmospheric conditions of 20.0 ± 0.1 °C and 65 ± 2% relative humidity were consistently maintained throughout the experiment.

2.2.2.4 Thermal stability. The thermogravimetric analysis (TGA) of composites was conducted using a thermal analyzer (SDT650, TA-Instruments, USA) according to ASTM E-1131-20 standard. During the experiment, a 400 mg of each composite specimen was smashed into small fragments using a mortar and then 15 mg was transferred to an alumina crucible for testing. The study was conducted under nitrogen atmosphere at temperatures between 25.6 °C to 500 °C, and the increase of heat was kept 10 °C per minutes. The mass loss depending on temperature and the resultant residue yield at specific degradation temperatures was recorded and presented accordingly.

The differential scanning calorimetry (DSC) studies concerning the temperature-depended transformations of composites were observed using the same instrument (SDT650, TA-Instruments, USA) at 25.6 °C to 500 °C with nitrogen flux of 20 mL min^{-1}. The degree of crystallinity of composites from DSC analysis was determined following eqn (5).

$$\text{Crystallinity index } X_c = \frac{\Delta H_f}{\Delta H_f^0} \times 100 \quad (5)$$

ΔH_f = heat of fusion of the developed composite, ΔH_f^0 = heat of fusion of 100% crystalline PP (ΔH_f^0 = 207 J g^{-1}).[27]

3 Results and discussion

3.1 Mechanical properties

The mechanical characteristics of the composite specimens were evaluated by analyzing tensile, bending and impact strength. Tensile strength refers to the highest load that a material can endure under continuous stress, while flexural strength denotes a material's capacity to bear bending loads. Several important factors, including fiber selection, the orientation of reinforced fiber within the composite, and fiber volume proportion, primarily influenced the strength of composites.[7] Additionally, the matrix and its adhesion with fiber, processing techniques, ambient conditions, and post-processing treatments also significantly persuade the composites' strength (Table 3).

Fig. 2 illustrates the tensile and bending characteristics of cotton–PP composites developed by uniform blending of fibers. Among the composites, the specimen CPP2 with 40.0% cotton fiber showed the highest tensile strength and modulus of 62.09 MPa and 1.37 GPa, respectively (Fig. 2a and b). The maximum tensile strength of CPP2 was attributed to the intimate blending of cotton and PP fibers at their optimum volume fraction. Because of uniform blending, the reinforced fiber had mixed consistently with matrix polymers, which created scope

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Table 3 Comparative tensile and flexural properties of the composite CPP2 and the similar composites developed in earlier studies

	Tensile properties			Flexural properties			
Fiber proportion	Strength (MPa)	Modulus (GPa)	Elongation (%)	Strength (MPa)	Modulus (GPa)	Elongation (%)	Ref.
40%	**62.09**	**1.37**	**14.15**	**138.90**	**5.15**	**8.91**	**This study**
48.70%	45.0	3.0	—	105.0	0.119	—	28
60%	48.0	0.451	—	—	—	—	29
40%	53.6	3.00	—	—	—	—	30
30%	28.07	1.87	18.01	45.3	1.92	—	31

for robust bonding between them at their interface and, as a result, demonstrated the highest tensile strength.

A gradual decrease in tensile strength with increasing cotton fiber in the composites was observed in the current study. Consequently, the composite CPP3 and CPP4 with higher amounts of reinforced fiber showed an average tensile strength of 54.95 and 45.96 MPa, respectively. In the case of tensile modulus, a similar trend was also observed for CPP3 and CPP4 with the values of 1.19, and 0.55 GPa, respectively (Fig. 2b). Due to the higher volume fraction of reinforced fiber in materials, the polymer matrix had a limited scope of adequately attached with the fiber. Accordingly, composites with the high volume of fibers, there is expected to be higher fiber–fiber interaction compared to fiber–matrix interaction, resulting in a decrease in strength of composites. Therefore, the elongation at break of composites was also increased with the percentage of reinforced cotton fiber, as shown in Fig. 2a.

Regarding flexural strength, the developed composites exhibited a similar pattern to tensile strength. The specimen CPP2 exhibited a maximum flexural strength of 138.90 MPa and a modulus of 5.15 GPa (Fig. 2c and d). This high performance can be attributed to the same underlying reason as stated for the tensile strength. Similarly, with increasing the fiber volume fraction, the composites demonstrated a decrease in flexural strength with values of 130.03 and 102.2 MPa for CPP3 and CPP4, respectively.

Among the developed composite, the composite CPP2, with the optimum fiber and matrix ratio, exhibits considerably higher tensile and flexural performances compared to cotton–PP composites developed in earlier studies using different

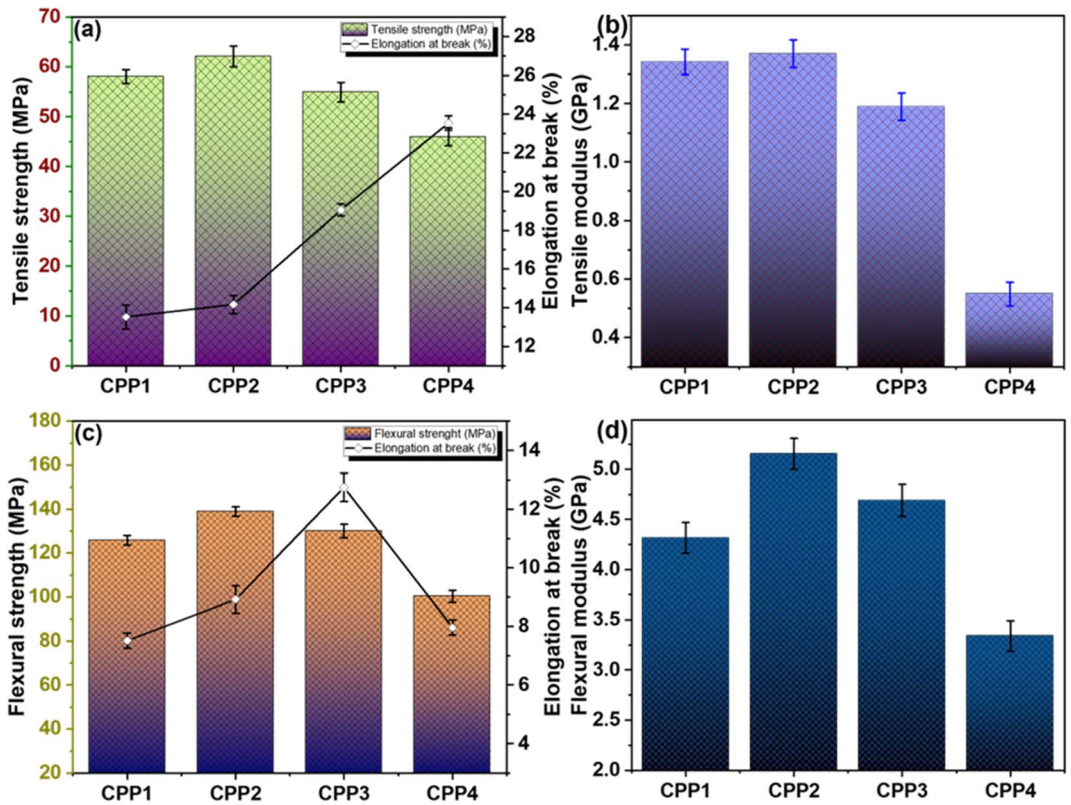

Fig. 2 Mechanical properties of developed cotton-reinforced PP composites; (a) tensile strength and breaking extension, (b) tensile modulus, (c) flexural strength and breaking extension and (d) flexural modulus.

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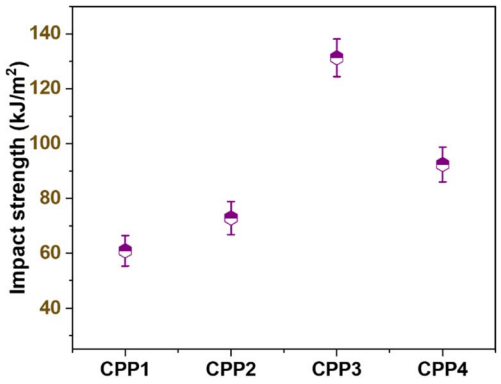

Fig. 3 The change in the impact strength of composites with the amount of reinforced fiber in the material.

techniques. Cotton–PP composites developed by Fares *et al.*[28] using a thermoforming pressing technique showed the highest 45.0 MPa and 105.0 MPa tensile and flexural strength, respectively, for the material with 50.0% reinforced cotton fiber. Lin *et al.*[29] found a maximum tensile strength and modulus of 48 MPa and 0.451 GPa, respectively, of cotton–PP composites developed through stacking method with cotton and PP ratio of 45/55. Serra *et al.*[30] showed the maximum tensile of 53.6 MPa can be achieved for cotton–PP composites developed by injection molding technique. Rukmini *et al.*[31] found 28.07 MPa and 45.3 MPa of tensile and flexural strength, respectively, for the composites with 30% reinforced cotton fiber developed using the compression molding technique.

The impact strength of the developed composites in Fig. 3 shows a positive correlation between fiber volume fraction and impact strength, suggesting the rise in fiber content enhances the energy absorption capability of the composite up to a certain point. Materials with high impact strength effectively absorb energy when subjected to sudden forces, and generally, lower interfacial bonding can result in higher impact resistance due to delayed crack propagation.[32] Among the tested composites, CPP3 exhibited the highest impact strength of 130.75 kJ m^{-2} (Fig. 3), which is 2.15 and 1.96 times greater than CPP1 and CPP2, respectively. The superior performance of CPP3 is attributed to the optimized fiber-to-matrix ratio, which facilitates effective stress transfer within the composite. Conversely, CPP1 and CPP2 exhibited lower impact strength, likely due to the higher matrix content, which promotes stronger interfacial bonding and increased friction stress between the fiber and matrix. While strong fiber–matrix interactions contribute to structural integrity, they may also reduce material toughness, resulting in sudden failure under impact loading.[33]

The composite CPP4, however, demonstrated an impact strength of 92.42 kJ m^{-2}, which is higher than CPP1 and CPP2 but lower than CPP3. This reduction in impact strength compared to CPP3 can be explained by excessive fiber content, which leads to increased stress concentrations and interfacial voids. The existence of such defects may serve as initiation points for crack propagation during impact, thereby compromising the composite's ability to absorb energy efficiently. These findings suggest that an optimal fiber-to-matrix ratio is critical for maximizing the impact resistance of cotton–PP composites developed through the intimate blending of fibers.

3.2 Morphological analysis

The morphological examination employing a scanning electron microscope (SEM) in the present study was conducted to observe the arrangement of fiber components in the preform and the modes of mechanical failure of composites due to tensile stress. The SEM images in Fig. 4 displayed the blended fibers preform and fractured cross-sectional view of the developed composite after tensile failure. The typical convolution of natural cotton fibers and the rodlike shape with the regular surface of PP fibers in the SEM image of Fig. 4a shows the presence of cotton and PP fibers in the composite preform. Besides, the uniform distribution of cotton and PP fibers in the carded lap (Fig. 4a), confirms the uniform mixing of fiber components in the composite. The fractured cross-section of the composite due to mechanical failure in Fig. 4b revealed a complex internal structure with randomly arranged fibers, including fiber breakage/fracture and matrix cracking. The developed composite exhibited improved interfacial adhesion, reduced fiber pull-out, and clean fracture surfaces. No

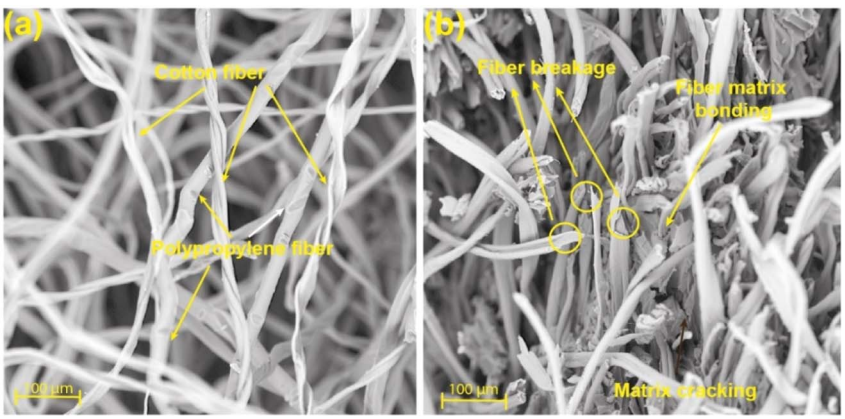

Fig. 4 SEM micrographs of cotton-reinforced polypropylene composite; (a) the distribution of cotton and PP fiber in the composite preform before consolidation and (b) fractured cross-sectional view of consolidated composite (CPP2) after tensile failure.

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interfacial delamination was observed between the reinforcement and matrix upon fracture. Instead, they displayed a cohesive ribbon-like structure comprising interlocked fiber–matrix bundles formed by reinforced fibers and polymers. This interconnected and resilient fibrous bundle or ribbon likely reduces the occurrence of fiber pull-out or delamination during fracture events, as it effectively distributes applied forces across the composite structure, leveraging the strength of the fibrous network. The observations suggest good bonding between the cotton fibers and the polypropylene polymer matrix due to the intimate blending.

3.3 Heat barrier properties

Thermal conductivity is the ability to transfer heat through the solid material, which is an indicator of the heat insulation characteristics of composites. The heat conductivity of NFRCs principally depends on the types of fiber used, the presence of air space, the matrix polymer, the interfacial interaction, the traveling distance of heat, and the temperature difference between the two surfaces of composites.[7]

Fig. 5a illustrates the thermal conductivity and conductive heat resistance of four different cotton-reinforced PP composites developed through consistent blending of fibers. All the composites demonstrated low thermal conductivity owing to the lower thermal conductivity of cotton fiber.[34] Among them, CPP4 with 60.0% cotton fiber showed the lowest thermal conductivity of 0.063 W m^{-1} K^{-1}. This low heat conductivity of composites can be attributed to a higher proportion of reinforced fiber in the composite and the inherent structural properties of cotton fiber, including the presence of lumen that forms air pockets inside the fiber structure.[35] Additionally, the increased reinforced cotton fiber leads to reduced interfacial attachment with higher voids in the composite, which creates air pockets, consequently, a longer traveling distance for transferring conductive heat through the material. However, the presence of a polymer matrix in the composite adversely affects the thermal resistance, as polypropylene has a thermal conductivity 0.22 W m^{-1} K^{-1}.[36] The thermal conductivity of composites increases proportionally with the increase in the amount of polymer matrix. With the increase of matrix polymer, the interfacial bonding strengthens, and the gap between fiber and polymer decreases, resulting in the fast conduction of heat through the composites.

In terms of resisting the flow of heat through the structure, thermal resistance increases with increasing the cotton fiber in the developed composites (Table S1†). The specimen CPP4

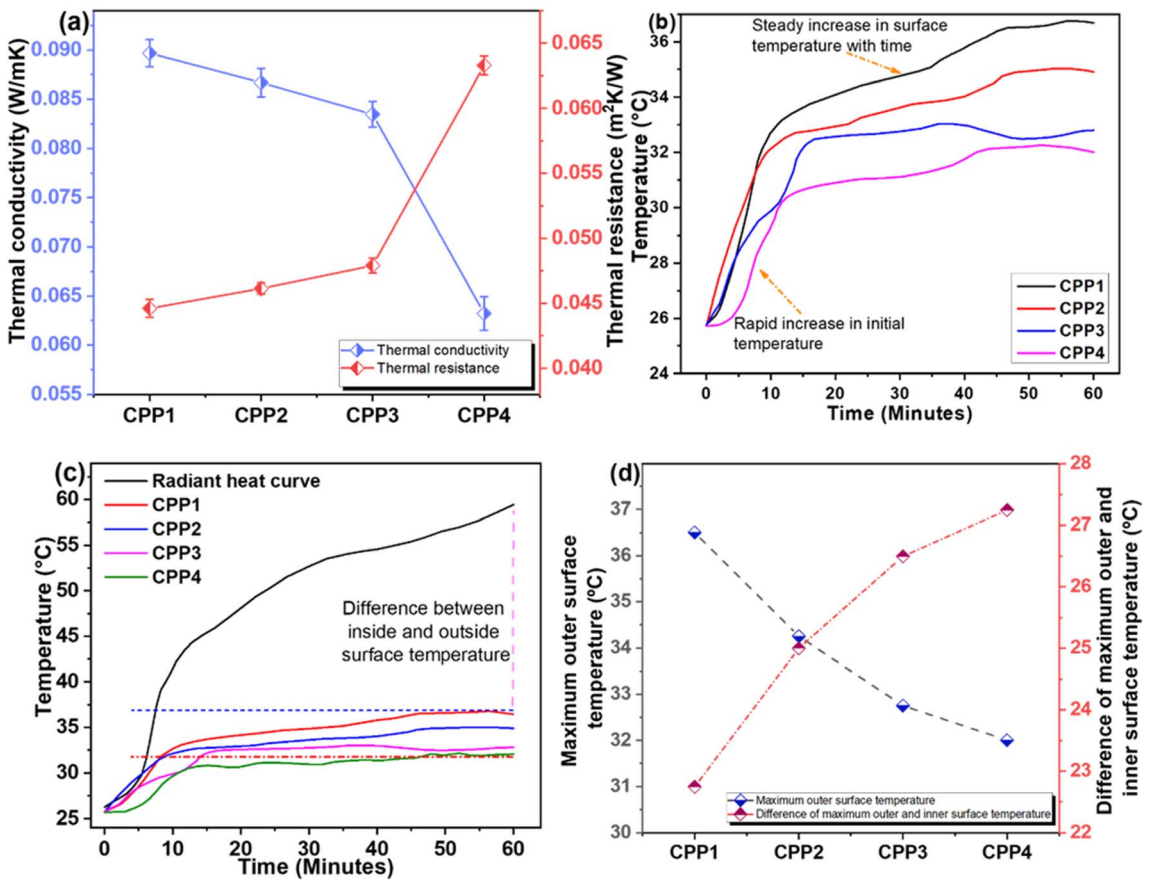

Fig. 5 Analysis of heat resistance performance of fabricated composites; (a) thermal conductivity and resistance to conductive heat transfer, (b) the rise in composites outer surface temperature with time after exposure to radiant heat, (c) temperature curve with the difference in inner and outer surface temperature exposed to radiant heat, and (d) maximum outer surface temperature and difference between the maximum inner and outer surface temperature of developed composite specimens.

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demonstrated superior thermal resistance and showed 43.17% higher thermal resistance compared to CPP1 (Fig. 5a). In this context, the low-thermal-conductive cotton fiber serves as a thermal barrier, effectively impeding heat transfer by creating a separation between the thermally conductive polypropylene matrix. This separation results in a reduction of heat transmittance within the composites.

The time–temperature graphs shown in Fig. 5b demonstrated the composite's function when exposed to a radiant heat source. An increment in the surface temperature of all specimens was observed over a period of time. The cotton–PP composites, particularly CPP4, demonstrated exceptional resistance to radiative heat. This was evident from the lowest surface temperature of 32 °C (Fig. 5d). The improved performance can be ascribed to the structural characteristics of cotton fiber, as previously mentioned, which effectively limit the conduction and convection of radiant heat due to its low thermal conductivity (TC). In addition, the reinforced cotton fiber has a larger specific surface area due to its fineness (8 to 20 microns), which enhances the scattering of heat from the material's surface.[37] Consequently, the composites experience a reduction in heat transfer.

The difference between the outer and inner temperatures is shown in Fig. 5c and d. The difference in temperature between the inner surface and outer surface of the composites after being exposed to a radiant heat source for about 60 minutes is 22.74 °C, 24.99 °C, 26.49 °C, and 27.24 °C, respectively, for CPP1, CPP2, CPP3, and CPP4 composites. The developed cotton–PP composites showed a gradual rise in temperature difference with increasing fiber content in composites. Accordingly, CPP4 composites with the highest proportion of cotton fiber demonstrated the highest temperature difference due to low thermal conductivity of cotton fiber.

3.4 Thermal imaging

Thermal imaging, using a thermographic infrared camera, was employed to verify the thermal insulation properties of cotton fiber-reinforced PP composites after placing them on a hot surface with constant temperature. The surface temperature of the developed composites, after fixed intervals, was captured and presented in Fig. 6.

The developed cotton–PP composites with a higher volume fraction of reinforced cotton fiber showed a slow increase in surface temperature. Accordingly, specimen CPP4 showed the

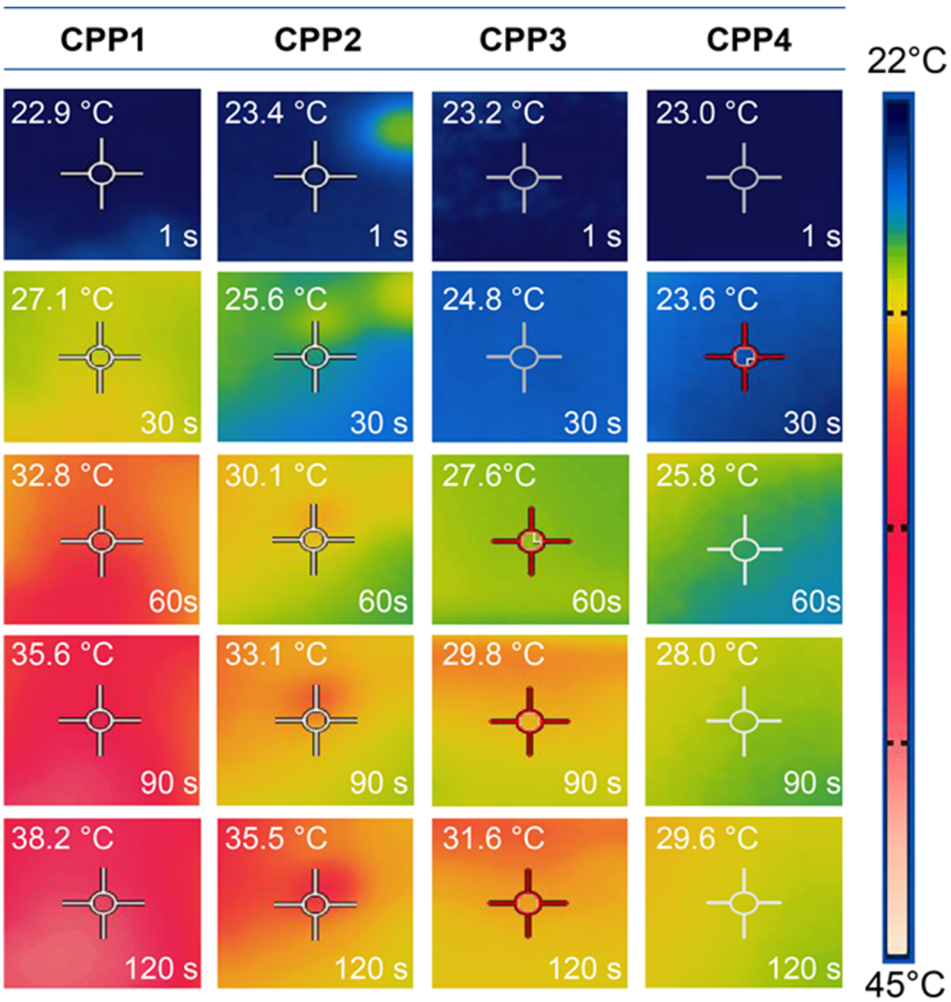

Fig. 6 Infrared thermal images of cotton–PP composite specimens of varying amount of reinforced fiber materials.

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slowest increase in temperature of 23.0 °C, 23.6 °C, 25.8 °C, 28.0 °C, and 29.6 °C at 1 s, 30 s, 60 s, 90 s, and 120 s, respectively. On the contrary, composite CPP1 showed a comparatively higher increase in temperature of 22.9 °C, 27.1 °C, 32.8 °C, 35.6 °C, and 38.2 °C at 1 s, 30 s, 60 s, 90 s, and 120 s, respectively. The specimen CPP4 demonstrated a much lower surface temperature than CPP1 at the end of the test, which was attributed to the low thermal conductivity of cotton fiber, as discussed earlier. There was also a negative impact of high heat conductive PP on the thermal barrier performance of composites since increasing polymer material results in good bonding of fiber and matrix, and the heat transmission due to conduction was increased.

3.5 Thermal analysis

The thermal analysis of developed composite materials was executed using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) to evaluate their ability to absorb heat energy after exposure to high temperatures. The TGA was used to determine the materials' thermal properties and breakdown characteristics along with the decomposition rate of materials during heating processes. This data provides insights into the material's thermal stability, optimal temperature for use, and suitable environmental conditions.[38] Data obtained from the TGA study of developed cotton–PP composites are plotted and presented in Fig. 7a.

Since all test specimens consist of similar fiber and matrix, they demonstrated almost comparable trends in the curve. TGA curves in Fig. 7a exhibit three-phase degradation of developed composites exposed to high temperatures. The initial stage of mass reduction is linked to the vaporization of moisture or water contained within the composites around the temperature reaches approximately 100 °C. Then, the degradation of cellulosic material in cotton fiber occurred at a temperature range between 255 and 430 °C.[39] Cellulose decomposition typically begins with an intra-molecular reaction that eliminates water and forms levoglucosan.[40] This was followed by the formation of shorter molecules by depolymerization of the cellulose macromolecules. Additional reactions might take place, including the breaking of glycosidic linkages (C–H, C–O, C–C) and the removal of water molecules with the elimination of carboxyl and carbonyl groups.[41] The significant degradation of polypropylene was also completed between 400 and 500 °C since the decomposition of polypropylene generally occurred at around 463 °C.[42] The final residue (%) of composites collected at 475 °C during the investigation showed an increased amount of residue with increasing the fraction of cotton fiber, and the highest residue of 16.39% was found for CPP4 (Table 4).

DSC test was employed to evaluate the heat absorption and release properties, phase change temperature, and viscoelastic properties of composites, which primarily depend on the specific characteristics of the fiber and polymer and their attachment at the interface. Different exothermic and endothermic peaks of composites were identified from the DSC analysis, where exothermic peaks specified oxidation, chemical reactions, and decomposition, and endothermic peaks

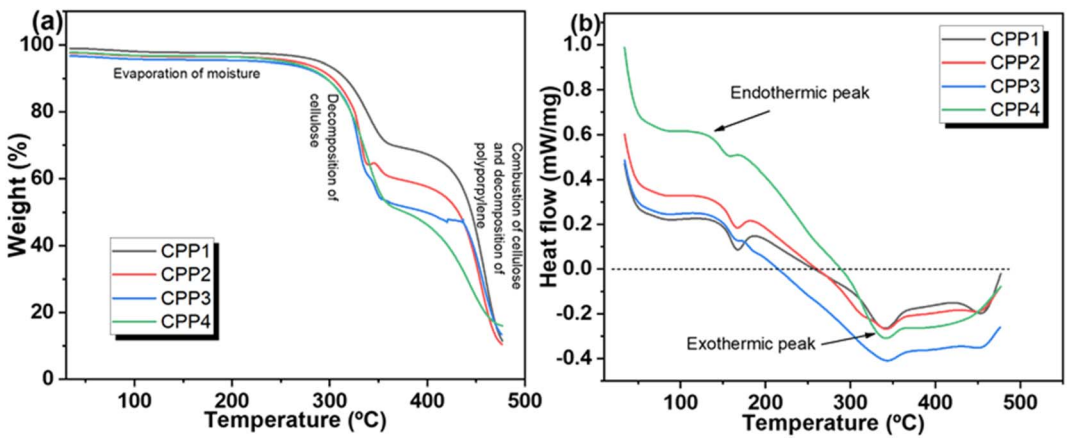

Fig. 7 Thermal analysis of test specimens at high temperatures; (a) thermogravimetric analysis (TGA) and (b) differential scanning calorimetry (DSC) curves of developed cotton-reinforced polypropylene composites.

Table 4 Residue (%), endothermic and exothermic peak, heat flow, and crystallinity index (%) of cotton–PP composites obtained from TGA and DSC curve

Sample	Residue (%) at 475 °C	Endothermic peak (°C)	Exothermic peak (°C)	Heat flow ($J \cdot g^{-1}$)	Crystallinity index (%)
CPP1	10.42	186.11	341.20	73.02	35.27
CPP2	11.99	181.91	342.83	79.44	38.37
CPP3	13.58	174.55	342.83	59.88	28.92
CPP4	16.39	168.82	342.32	43.44	20.98

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Table 5 ANOVA test of tensile, flexural, and thermal insulation properties[a]

			Tensile properties			Flexural properties		
	TC	TR	Strength	Modulus	Strain	Strength	Modulus	Strain
F value	222.6	169.94	23.02	41.22	17.12	29.19	3.17	11.96
P value	0.000066	0.0001	0.0003	0.000032	0.0008	0.0001	0.0852	0.0025

[a] TC = thermal conductivity and TR = thermal resistance.

indicated melting, dehydration, phase transition, and crystallization of composites.[43]

Fig. 7b depicts the DSC curves of cotton-reinforced composites, which showed a similar trend for all specimens in exothermic and endothermic peaks. The endothermic peak was noticed between 150 to 210 °C, signifying the thermal stability of cotton–PP composites at elevated temperatures. The exothermic peak, on the other hand, was detected between 330 to 390 °C, suggesting the thermal degradation of cellulose, the primary constituent of cotton fiber. Additionally, the crystallinity index of composites, to understand the bonding behavior of matrix and fiber components, was calculated from the DSC curve. The composite CPP2 exhibited the highest crystallinity index of 38.37%, indicating the formation of robust bonding between the reinforced fiber and polymer at their optimal volume fractions (Table 4). This also correlates with the highest tensile and flexural strength of the same composite, as discussed previously. A decrease in crystallinity indices with increasing the amount of reinforced cotton fiber in the composite, suggested the reduced bonding strength between fiber and polymer, consequently, the lower mechanical strength of materials.

3.6 Statistical analysis

Tensile, flexural, and thermal insulation properties of composite specimens were statistically evaluated using analysis of variance (ANOVA) and Tukey HSD test at the 5% significance level ($\alpha = 0.05$). ANOVA results of tensile, flexural, and thermal insulation properties revealed significant differences among the developed composites except flexural modulus (Table 5).

From Tukey HSD tests, it was observed that for both tensile and flexural strength (Table S2†), there is a significant difference between specimens CPP2 and CPP4 ($p < 0.01$) and between CPP3 and CPP4 ($p < 0.01$). However, no significant differences between specimens CPP1 and CPP2 and between CPP1 and CPP3 were noticed. Tukey HSD was also conducted to check the significant differences in thermal conductivity of different composite specimens (Table S3†). It was found that there is a significant difference in thermal conductivity among the composite specimens of CPP1–CPP3 ($p < 0.0$), CPP1–CPP4 ($p < 0.01$), CPP2–CPP4 ($p < 0.01$), and CPP3–CPP4 ($p < 0.01$).

A quadratic regression analysis was done in order to understand the relationship between cotton fiber percentage, tensile strength, and thermal conductivity (Fig. 8). Here an independent variable is the cotton fiber percentage, and the dependent variables are tensile strength and thermal

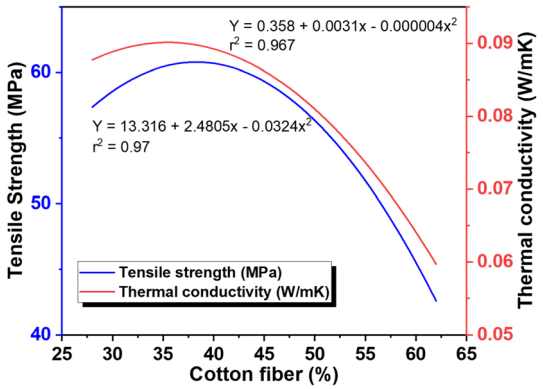

Fig. 8 Quadratic regression model of tensile strength and thermal conductivity of developed cotton–PP composites.

conductivity. From the statistical analysis, it was found that with the increase in cotton fiber percentage, thermal conductivity decreased significantly ($F = 222.6$, $p < 0.0001$) *i.e.*, thermal insulation properties increased significantly. However, with the increase in cotton fiber percentage, tensile strength also decreased significantly ($F = 23.02$, $p < 0.001$). As tensile strength and thermal insulation properties follow opposite trends with the increase of natural cotton fiber percentage, an optimum point was identified from the regression model (Fig. 8), which is 57.0% of cotton fiber, where both good thermal insulation properties (TC $\approx$ 0.07 W m^{-1} K^{-1}) and high mechanical properties (tensile strength $\approx$ 49.5 MPa) would be found.

4 Conclusions

The present study attempted the development of high-performance fiber-reinforced polymer composites through the uniform blending of natural cotton and PP fibers and investigated the optimization of mechanical strength while maintaining adequate thermal insulation properties for engineering applications. The mechanical properties of uniformly blended cotton–PP composites were evaluated by conducting tensile, flexural and impact strength tests. Among the developed composites, the specimen CPP2, with the cotton and PP ratio of 40/60, showed the highest tensile and flexural strength of ~62.09 MPa and ~132 MPa, respectively. The same material also displayed tensile and flexural modulus of ~1.37 GPa and ~5.15 GPa, respectively. The composite CPP2, because of sufficient interfacial adhesion between reinforced cotton and PP fibers at their optimal volume fraction, demonstrated the

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highest tensile and flexural strength among the composites. Beyond the optimum volume fraction of 40/60, a decrease in tensile and flexural properties was observed with the cotton fiber content in the materials, and the composites with higher amounts of cotton fiber exhibited a declined trend in tensile and flexural strength. In the case of impact strength, the composite CPP3, however, displayed the highest impact strength of 130.75 kJ m^{-2} due to absorbing higher energy before breaking.

Regarding thermal insulation properties, the composite CPP4, owing to the highest amount of cotton fibers, demonstrated the lowest thermal conductivity of ~0.063 W m^{-1} K^{-1} and the highest conductive heat resistance of ~0.063 $m^2 \cdot K$ W^{-1}. It also showed the maximum resistance to radiative heat transfer after exposure to radiant heat. This superior heat barrier performance of CPP4 containing the maximum amount of cotton fibers was primarily attributed to their porous structure and intrinsically low thermal conductivity. As the amount of cotton fiber in the composite increased, a noticeable upsurge in both the radiative and conductive heat resistance of materials was observed. The thermal imaging test of composites also revealed conclusive evidence of their consistent resistance to heat. Subsequently, the thermogravimetric study confirmed the adequate thermal stability of developed composites when exposed to elevated temperatures, especially any significant mass loss of materials observed up to 200 °C. The differential scanning calorimetry study revealed the viscoelastic properties of the composites, which showed the reduced crystallinity of the composite materials with the increase of fiber loading in PP polymer.

Based on the outcomes of the current study, it can be concluded that fiber-reinforced polymeric composites developed by uniform blending of natural cotton and PP fibers with enhanced interfacial adhesion possess sufficient mechanical strength and adequate thermal barrier properties. Thus, they have the potential to be employed as engineered materials in diverse fields, including automotive, railway, aircraft and building, where both mechanical and thermal barrier performances are the key requirements.

Ethical statement

This article does not involve any research on human participants or animals.

Data availability

The authors declare that all the data generated or analyzed during this study are available within the article.

Author contributions

M Abu Darda: methodology, investigation, writing – original draft. M A Rahman Bhuiyan: conceptualization, supervision, investigation, writing – original draft. M Ashnaim Bari: methodology, investigation. Shafiqul Islam: writing – review and editing. M Jakir Hossen: methodology, investigation.

Conflicts of interest

The authors declare no competing interests.

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