



Experimental Investigations of Flammability, Mechanical and Moisture Absorption Properties of Natural Flax/NanoSiO₂ Based Hybrid Polypropylene Composites

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Abstract

Natural fibre-reinforced polymer composites, recently developed, provide several socioeconomic and sustainability benefits for a wide range of technical applications. The purpose of this study was to look at how natural flax fibre (30 wt%) and silicon dioxide (SiO₂ (0, 2.5, 5, 7.5, 10, 12.5 wt%)) affect ternary mixes' mechanical, combustible, and water absorption qualities. A cone calorimetry investigation revealed that the inclusion of nanosilicon dioxide might reduce the polypropylene (PP) composites made from flax fibre's total heat release, total smoke release, and heat release rate. Using 10 wt% nanosilicon dioxide, the heat release rate (255 kW/m² to 161 kW/m²) was considerably reduced. The mechanical characteristics of the composites increased with the addition of nanosilicon dioxide, and the flexural (50.97 MPa) and tensile strength (42.65 MPa) of the materials reached their maximum values at 10 weight% nanosilicon dioxide. Nanosilicon dioxide exhibited homogeneous dispersal in the flax fibre-based PP composites, with an increase in nanosilicon dioxide from 2.5 to 10 wt%, according to scanning electron microscopy observations on the fractured portion of the hybrids. The lack of a recognisable peak in the X-ray diffraction (XRD) after the integration of nano-SiO₂ particles suggests that the granules were homogeneous. The highest water absorption in flax/PP hybrids decreases by 13.25, 15.69, 20.16, 23.63 and 19.52% as compared to pure PP and PP with flax fibre with the addition of 2.5, 5, 7.5, 10 and 12.5 wt% of SiO₂ filler, respectively. Based on the obtained results in natural flax fibre-based hybrid composites, the nanoSiO₂ particles worked as barriers to prevent moisture absorption and increase their rigidity. These outcomes imply that such materials would be appropriate for use in moist settings, like those seen in the maritime, outdoor, and packaging sectors. Additionally, these results imply that such composites may find use in sectors where fire safety is a top priority.

Keywords Nano SiO₂ · Flame retardancy · Flax fiber · Tensile strength · Nanocomposites · XRD analysis

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1 Introduction

Fibre based polymer materials are extensively used in a variety of sectors, including aviation, automobiles, defence, manufacturing, maritime, and athletic products, due to their improved physical and mechanical characteristics [1]. Natural fibres have been replacing synthetic fibres (particularly glass fibres) in recent times due to benefits such as biological degradation, low density, sustainability, inexpensive prices, and strong mechanical qualities [2, 3]. Banana, flax, kenaf, coir, bamboo, cotton, bagasse, oil palm, sisal, and hemp are examples of natural fibres. Natural fibre composites have been widely used in a variety of industries, including building, space vehicles, automotive interior linings, and even athletic products. As a result of its reduced impact on the environment as well as its price relative to typical carbon

or other synthetic fibres, flax fibre has gained popularity as a reinforcement made from fibres among the natural fibres [4]. In the past few years, flax fibre has become a popular plant fibre in thermoplastic-based hybrids. Still, flax-fibre-reinforced plastic materials' limitations, like limited strength and hydrothermal characteristics, restrict their potential, particularly in building elements [5]. Natural-fibre-reinforced polymer (NFRP) composites are currently confined to non-structural or frame uses, such as vehicles and aeroplanes, while constructing interior designs. The fundamental disadvantage of using flax fibres as supplements in hydrophobic matrices of polymers is their hydrophilic character. Chemical or physical approaches have been employed to change the fibre surface to enhance the mechanical behaviour of natural composites [6]. The greatest flaxseed-producing area on the globe is Asia-Pacific. The main manufacturers and consumers of flax seeds in this area are the Republic of Kazakhstan, India, China, Australia, and Baghdad. In accordance with FAOSTAT (Food and Agriculture Organization Corporate Statistical Database), Kazakhstan alone will have around 1.34 million acres of produced land in 2020, or roughly 37% of the entire produced region. During the past several years, flaxseed's prospects for export have expanded in response to the rising worldwide demand for it. Kazakhstan's linseed exporters rose in value by more than 50% between 2017 and 2021, achieving USD 226 million in 2021 [7, 8]. The Asia-Pacific area consumes the most flaxseed owing to the existence of densely populated, developing nations like China and India. Due to rising disposable incomes and an increase in middle-class families in the area over the past several years, it is projected that demand will continue to climb. Therefore, it is anticipated that the flax seed industry will be

driven by rises in consumption rates and financial freedom in the Asia-Pacific region. Approximately 7% of the quantities produced have been considered fibre waste in prior studies [9, 10]. The statistical evaluation of flax fibre production as well as waste consumption is displayed in Fig. 1. Based on the previous literature, some common properties of natural flax fibre were mentioned in Table 1.

One of the most promising approaches for improving the mechanical characteristics of fibre composites is the inclusion of nanomaterials. Two approaches were utilised in this respect: (i) dispersal of nanofillers within resin and (ii) growth or transplantation of nanofillers onto the fibre surface [12]. Nanotechnology advancements are accelerating research in areas such as materials, medicine, space science, electronic components, storage materials, and the

Table 1 Some Mechanical, Physical properties with Chemical compositions [11]

Sl.No	Particulars	Values
1	Density (g/cm ³)	1.42–1.56
2	Tensile strength (MPa)	340–1042
3	Elongation at break (%)	2.63–3.25
4	Young's Modulus (GPa)	27.42
5	Relative strength (mN/Tex)	430–520
6	Water Content (%)	5.36–7.26
7	Cellulose (%)	72–76
8	Hemicellulose (%)	18.20–21.03
9	Wax (%)	1.76
10	Pectin (%)	2.12
11	Lignin (%)	2.23

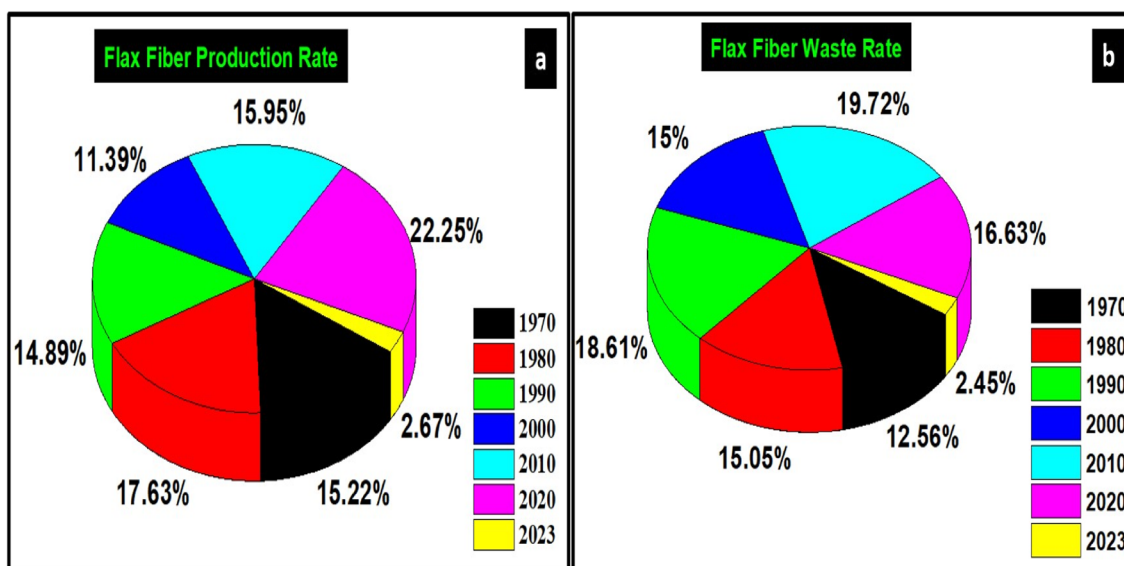


Fig. 1 Statistical analysis of **a** Flax fiber production rate; **b** Flax fiber waste rate

natural world. Small amounts of nanofillers are causing significant modifications and enhancing material effectiveness and characteristics [13, 14]. Recently, there has been a lot of attention paid to using regenerative and environmentally friendly materials that are naturally sustainable without sacrificing the standard of the initial resources [15]. The incorporation of 2.5 wt% salinized TiO₂ enhanced the mechanical properties of the organic fibre/epoxy composites by 43%, according to Zaer-Miri et al. [16]. Ashori and Nouabksh [17] created PP-based materials strengthened with wood flour using an injection moulding procedure and discovered that at 8% montmorillonite clay loading, durability improves by 21%, flexibility improves by 15%, and water consumption decreases by 4%. Hosseini et al. [18] employed SiO₂ filler materials with 3 and 7% weight proportions for the creation of polyethylene-based lightweight materials strengthened by bagasse fibre and found an 82.56% improvement in tensile properties in SiO₂-filled nanocomposite composites. Wang et al. [19] investigated the influence of clay-coated flaxseed fibres on epoxy materials. The altered natural fibre-based specimens enhanced bending and tension properties by 22% and 16%, respectively, as compared to the clean flax fibre specimen. Kushwaha et al. [20] investigated the impact of the incorporation of carbon nanotubes. They concluded that epoxy-based lightweight materials could be strengthened with bamboo fibres using a normal hand layup approach and discovered an 8.21% improvement in tension and a 4.3% rise in bending, as well as a drop in moisture absorption from 31.30 to 22.36%. Kumar et al. [21] demonstrated that the tension and bending behaviour of bamboo/epoxy-based hybrid laminates containing 5% nanoclay were increased by 42% and 26%, respectively, over plain composites.

Natural fibres added to polymeric materials may alter the mechanical characteristics of polymer assemblies. Aside from the primary characteristics of structure substances, mechanical attributes, and expense of materials, combustibility is a significant consideration for numerous uses that often limit the use of materials to the construction sector and automobiles [22]. A number of investigations have been conducted to date on the interfacial effectiveness of natural fibre-strengthened polymeric composites. Another important consideration is fire behaviour. Fire-resistant substances are required for security purposes [23]. PVC materials are a soul-extinguishing substance, although they ought to be prevented owing to the volatility of the gases produced. Polyolefins, another common monomer used in natural composites, combust and leak in the event of a fire, creating a dangerous situation. As a result, fire-retardant chemicals must be used to enhance fire behaviour [24]. Several studies on the fire effectiveness of composite materials reinforced with natural fibres

have been conducted. Kiguchi and Stark [25] conducted the study on fire resistance in wood-based composites. The authors create safe composites, and the flame-retardant ingredient ought to be chosen from phosphorous and artificial systems like hydroxide stagnates and borates. Borate chemicals were utilised, and the findings revealed that fire effectiveness improved. Schartel et al. [26] investigated the fire resistance of PP-based flax fibre. According to the scientists, the inclusion of n(OH)₂ and extensible graphene significantly changes the quantity of heat emission per mass loss.

Nevertheless, a few investigations on the fire-resistant performance of the natural fibre-strengthened polymers have been conducted [27]. Fire suppressing chemicals like phosphorus, halogen, and nitrogen were incorporated into the natural fibre and polymeric combination to enhance the fire behaviour of natural fibre-based lightweight materials. Because of their minimal toxicities, corrosive properties, and formation of smoke during manufacturing and combustion, nanomaterials like aluminium hydroxide, caly, and carbon nanotubes are now an emerging trend in the field of fire retardancy. One of these promising nanomaterials for flame retardancy is nano-silicon dioxide (nano-SiO₂). Nano-SiO₂ was commonly used as a polymeric filler to improve physical and thermal characteristics [28, 29]. Nano-SiO₂ has a larger effective surface area of 110–320 m²/g with an increased permeability of 90% when compared to microparticles. The primary goals of this study are to determine the mechanical and flammability characteristics of flax/SiO₂-based hybrid composites. To achieve the goals, the composites were made using the vacuum moulding process with varied weight proportions of flax and nanoSiO₂. The composites were mechanically (tensile, flexural, and impact), thermally (flammability) and moisture absorption evaluated after manufacture.

2 Experimental Works

2.1 Materials

The natural woven flax fibre (thickness of 0.50 mm and density of 1.61 g/cm³) was procured in Salam, Tamil Nadu, India. Granules of polypropylene (PP) with a melt flow index of 19.6 g.10 min – 1, melting and glass transition temperatures of 160°C and 100°C, respectively. For external diagnostic procedures, the Rithu chemical industries provide both polypropylene, sodium hydroxide (NaOH), and acetonitrile. The fire-retardant nano-SiO₂ with a diameter of 20 nm was procured from the Deekshitha chemical solution in Tamil Nadu, India. Table 2 demonstrate the

Table 2 Common mechanical properties of Natural flax and PP matrix [30]

Sl.No	Properties	Flax fiber	Polypropylene
1	Density (g/cm ³)	1.32	0.92
2	Moisture absorption (%)	7.26	Hydrophobic
3	Young's Modulus (GPa)	63.21	1.03–2.80
4	Tensile strength (MPa)	950	380
5	Elongation at break (%)	2.89	150–500

Table 3 Sample type and their weight proportions of Flax/PP/Nano-SiO₂ based hybrid Composites

Sl.No	Specimen Type	Flax fiber wt%	PP wt%	Nano SiO ₂ wt%
1	A	0	100	0
2	B	30	70	0
3	C	30	97.5	2.5
4	D	30	95	5
5	E	30	92.5	7.5
6	F	30	90	10
7	G	30	87.5	12.5

main properties of natural flax and PP matrix. The selection of nanoSiO₂ weight proportion based on the results of previous research [23, 31].

2.2 Alkaline Processing

Before each preparation or computation in the chemical alterations, flax fibres had been dehydrated at 50 °C for 70 h. Pre-dried fibres were submerged in aqueous solutions containing 5% NaOH at the temperature of the atmosphere. The fibres were steeped in the NaOH solution for 4 h before being thoroughly rinsed with water to remove any alkaline residue [32]. The modified fibres were then dried for 60 h in an air-flow oven at 50 °C. After 4 h, the washing was repeated with filtered water until the PH of the cloth was about 7. Finally, the neutralised flax fabric was placed on a suitable closed surface to avoid further absorption of moisture [33].

2.3 Composite Fabrications

To fabricate the composite materials, the vacuum backing method was employed. Condensed paraffin has been applied to a substance of about 300 × 300 × 3 mm³, enabling the specimens to be easily removed from the glasses. After that, the flax components were piled in various weight proportions and crudely cut to match the mould parameters. The polymers include 30% flax fibre by weight, with different weight proportions of nano SiO₂ (2.5 to 12.5 wt%). Table 3 displays the sample specifications and weight percentages. Figure 2 depicts the schematic representations of flax fibre/nano SiO₂ based hybrid composites. Figure 3 shows the fabricated composite plates

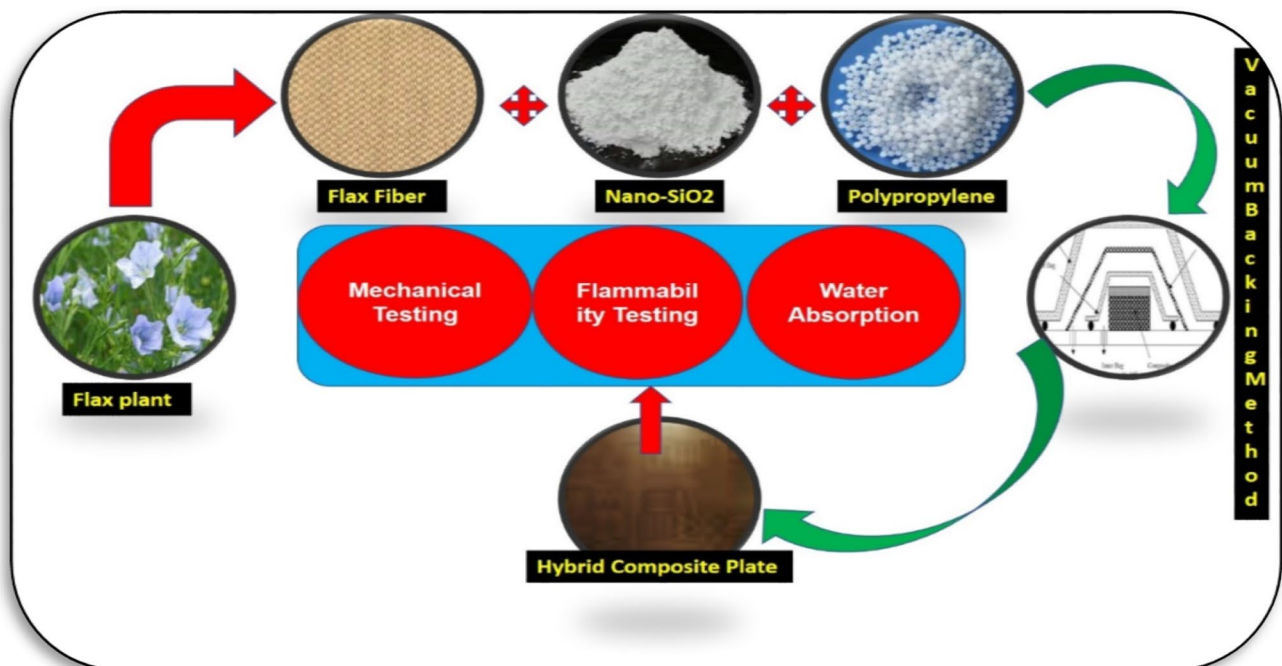
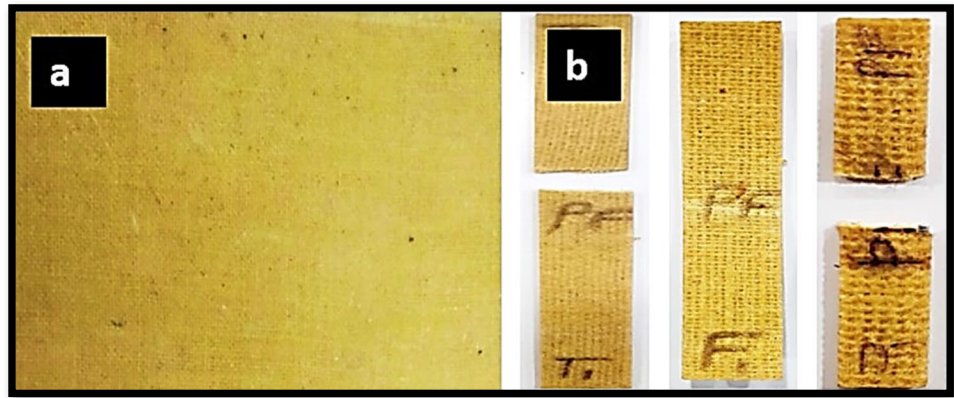
**Fig. 2** Schematic representation of fabrication of flax fiber/ nano SiO₂/PP based hybrid composites

Fig. 3 Flax/nano SiO₂ based hybrid **a** Fabricated laminate; **b** Failure of the specimen after tensile, flexural and impact testing



before and after testing. The following procedure explains the vacuum-backed composite fabrication method.

Vacuum Bagging Arrangement:

- (i). **Layup:** Set up the flax/nano SiO₂ hybrid prepreg layer on the releasing sheet or tool interface with the appropriate arrangement and order.
- (ii). **Vacuum Bag Placement:** Place a vacuum bag on top of the layup, making sure the edges are airtightly sealed.

Vacuum Application and Cure:

- (i). **Vacuum Application:** Remove the air from the vacuum bag by attaching it to a vacuum pump and generating a negative pressure condition.
- (ii). **Curing:** Apply pressure (10 Pa) and heat (70 °C) to the vacuum bagged layup while adhering to the proper curing cycle designed specifically for the polypropylene resin employed. To begin polymerization and obtain the requisite mechanical characteristics, a combination of temperature and time (15–60 min) is often required.

Cooling and Demoulding:

- (i). **Cooling:** Under regulated circumstances, gradually bring the cured laminate down to ambient temperatures in order to avoid undue thermal stress or deformation.
- (ii). **Demoulding:** With the utmost care, cautiously eliminate the dried laminate from the mould.

2.4 Composite Characterization

2.4.1 X-ray Powder Diffraction (XRD)

The crystal phase of the control fibres, alkali pre-treated flax fibres and nano-SiO₂ filled composites were shown by X-ray

diffractometer (Rikagu-9 Kw, Japan) with Cu K α radiation ($\lambda = 0.15406 \text{ \AA}$) and the 2θ range varies from 10 to 80 °. The test was conducted at Satyabhama University, Chennai, Tamil Nadu, India. Scanning electron microscope (SEM) (SUPRA 55, Carl Zeiss) was used to examine the fracture analysis of the hybrid composites after mechanical testing. SEM analysis also carried out under the same University, India.

2.4.2 Mechanical Testing

ASTM D 3039 was used to test the tensile properties, with a specimen dimension of $250 \times 25 \times 3 \text{ mm}^3$. The distance between both jaws, maximum speed, and gadget capabilities were all 3 cm, 0.3 cm/min, and 15 kN, respectively. The bending experiment was performed in accordance with ASTM D-790, and the sample size and the length within the supports were determined using the rule determined by the thickness of the specimen. The experiment speed was set at 2 mm/min. The ASTM D256 specifications were used to measure the impact properties of both unfilled and nano SiO₂ filled hybrid materials [34]. The examination employed a specimen dimension of $65 \times 12.7 \times 3 \text{ mm}$. A total of seven types of combinations were evaluated. All the experiments (for tensile, flexural, and impact) were repeated three times (totalling 63 specimens), and the mean results were provided.

2.4.3 Fire-Resistant Testing

The combustibility of the specimens was determined using an ISO 5660 cone calorimeter. A heat transfer of 60 kW/m^2 had been utilised externally. The cone-shaped calorimeter sample dimension was $100 \times 100 \times 3 \text{ mm}$. Each of the specimens was analysed horizontally, and the outermost framework was utilised for all testing. For computing, the flammable properties of the hybrid composites in all seven types of combinations were evaluated up to three times (A total of 21 samples), and the mean data was reported. Concerning those mean values, the standard

deviation was calculated. Concerning those mean values, the standard deviation was calculated. The Fig. 4 shows a sample image of flammability testing.

2.4.4 Water Absorption Behaviour

When wet or immersed in water, biological fibres and polymeric substances accumulate humidity. Water absorption experiments using ASTM D 570-98 were performed on multilayer polymers. The samples were 76.2 mm in length and 25.4 mm in wide. The samples underwent dehydration for 24 h at 60 °C prior to being refrigerated in a desiccator. The samples that had been created were fully immersed in a container of pure water held at 24 °C. After 120 min, each sample was cleaned with a clean cloth, and the weight was promptly recorded. The specimens were returned to the sea after being weighed. The identical technique was carried out at various time intervals of 24, 48, and 72 h. For each of the groups, a total of three experiments were repeated, and the mean values were used. The amount of moisture was calculated using the following formulas [35]:

$$\text{Water absorption} = \frac{W_2 - W_1}{W_1} \times 100 \quad (1)$$

Where W_1 and W_2 are the representations of the weight of the specimen before and after immersion in water.

3 Result and Discussions

3.1 XRD Analysis

Figure 5 displays the X-ray diffractograms for the flax that has not been treated with NaOH (Fig. 5a) and the flax that has (Fig. 5b). Figure 5 shows that for both normal and chemically modified fibres, there are two different maxima at lattice positions (1 1 0) and (2 0 0); consequently, vegetable

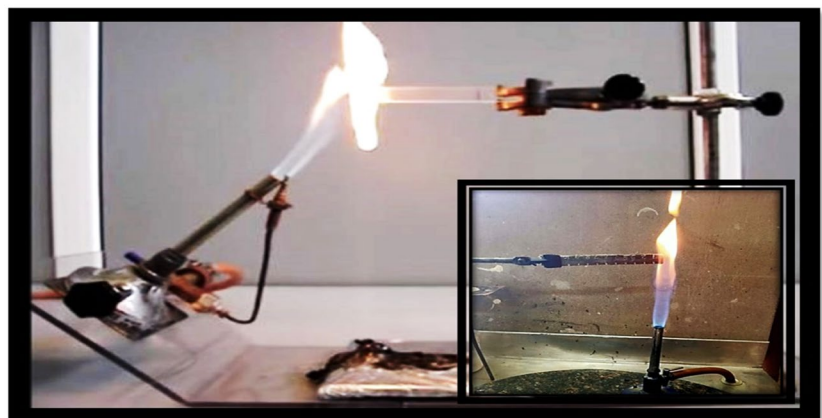
strands are the most prevalent source of both of these different peaks. They signify the existence of two key elements. Although a second strong peak (5 (b)) at $2\Theta = 21.53^\circ$ and 22.30° at lattice structure plane (2 0 0) indicates the existence of -Cellulose or Cellulose, the first peak (5 (a)) at $2\Theta = 16.10^\circ$ and 16.60° at lattice structure (1 1 0) indicates the existence of amorphous components of hemicellulose, cellulose, and others. pectin, lignin, etc. [27]. The blends of materials made from silicon dioxide and flax are depicted in Fig. 5c using XRD patterns. Figure 5c depicts an amorphous peak at $2\Theta = 22.2^\circ$, which is attributed to an amorphous SiO_2 structure in accordance with prior research. No further peaks can be seen, demonstrating the excellent purity of the produced SiO_2 . In accordance with earlier research, the generated SiO_2 is in a crystalline state for the XRD pattern of the SiO_2 created composite materials, which was connected to the SiO_2 crystallising a mixture of phases. As stated by JCPDS 00-082-1403, the spikes around 31.60 , 45.20 , and 56.72 are attributable to the (102), (202), and (104) structural planes, respectively. For example, the function of SiO_2 is to increase the durability and stability of the composite. This was enhanced by its crystalline shape [36].

3.2 Mechanical Properties

3.2.1 Tensile Properties

Figure 6 depicts the tensile characteristics of flax-PP-based lightweight composite materials. Flax fibre incorporation boosted polymeric materials tension properties by 30.8% but decreased the tensile elongation by 65.6% in organic fibres strengthened with polymeric materials, as expected. Figure 6 demonstrated that adding nano- SiO_2 to flax fibre-PP composites may boost their tensile strength. The materials with 10 wt% nano- SiO_2 exhibited maximum tension behaviour of 42.65 MPa, representing a 32% improvement over the materials without nano- SiO_2 . The tensile behaviour of flax fibre-PP composites altered by the incorporation

Fig. 4 Photographic image of horizontal flammability testing



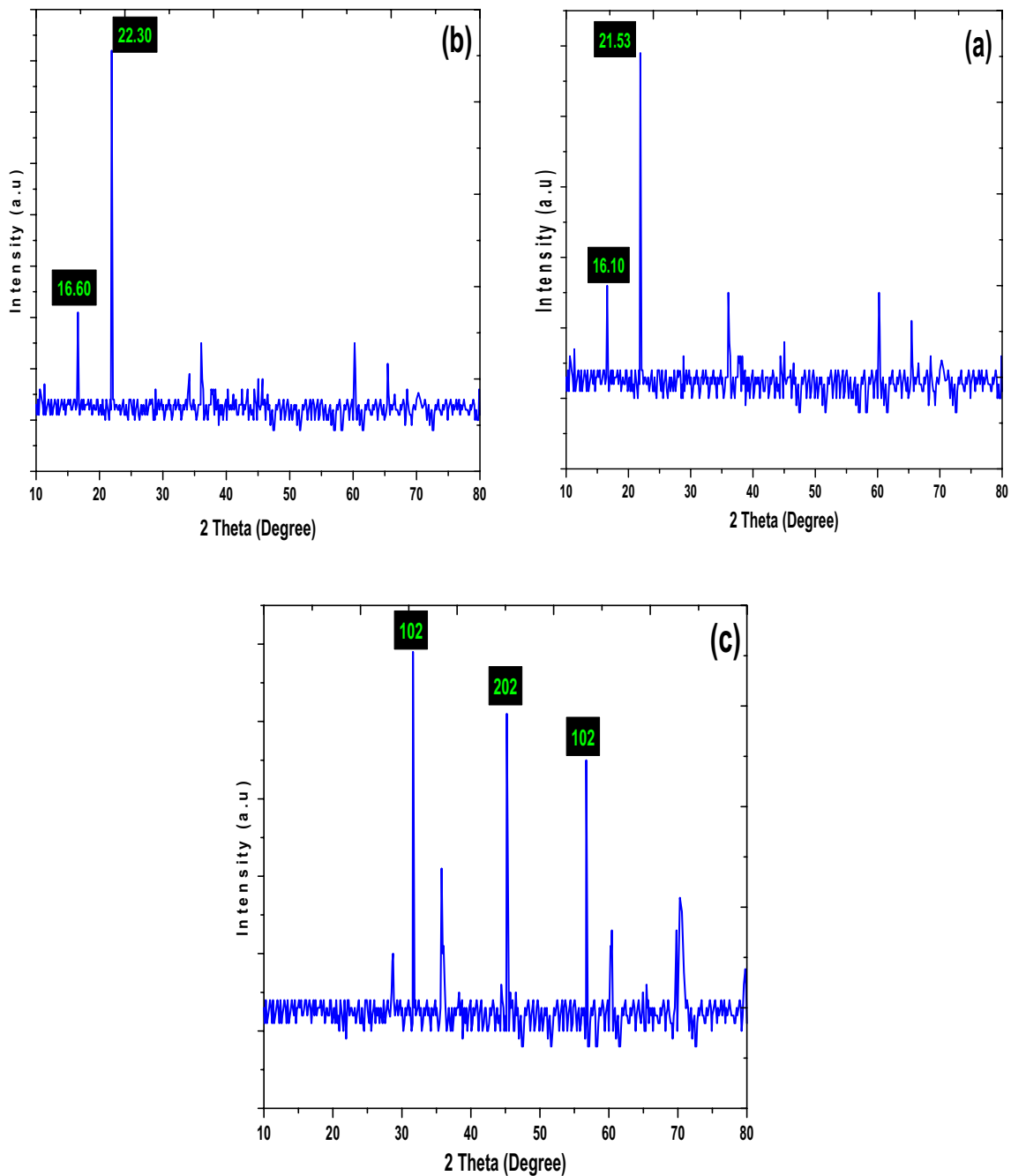
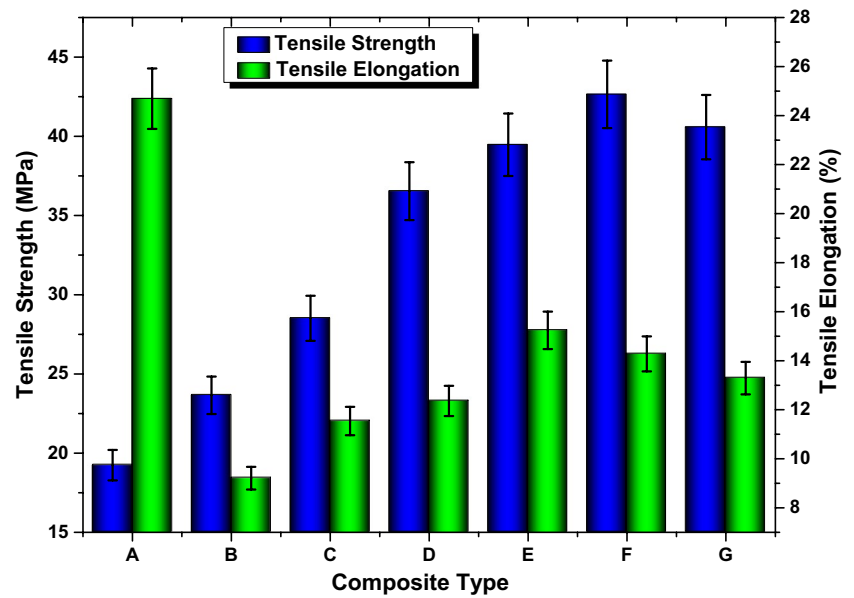


Fig. 5 XRD analysis of **a** Untreated flax fiber; **b** NaOH treated flax fiber and **c** NaOH treated flax/SiO₂ based hybrid composites

of nano-SiO₂ [37]. Increased tensile strength may derive from the fact that nano-SiO₂ has a greater rigidity than PP and flax fibre, resulting in improved flax fibre-PP materials strength. Additionally, the improved distribution of nano-SiO₂ in the hybrid composite, which facilitated an efficient transfer of stress as previously stated [31, 34], made this improvement in tensile strength achievable. Additionally, excellent bonding across their interfaces is caused by high interfacial bonding among alkali-treated flax fibre and

baseline PP matrices. Esterification, bonding of hydrogen, and polyamide production are potential interphase interactions between flax fibre and its base matrices. The combination of pretreated flax fibre with nanosio2 filler results in a material with excellent tensile strength. The tensile elongation of flax fibre-PP materials modified by nano-SiO₂ is shown in Fig. 6. Initially the tensile elongation values increased at 0 wt% of the nano-SiO₂. Furthermore, the elongation was decreased when the SiO₂ filler content

Fig. 6 Tensile strength and % of elongation on Flax fiber and different weight proportion of nano SiO_2 based hybrid composites

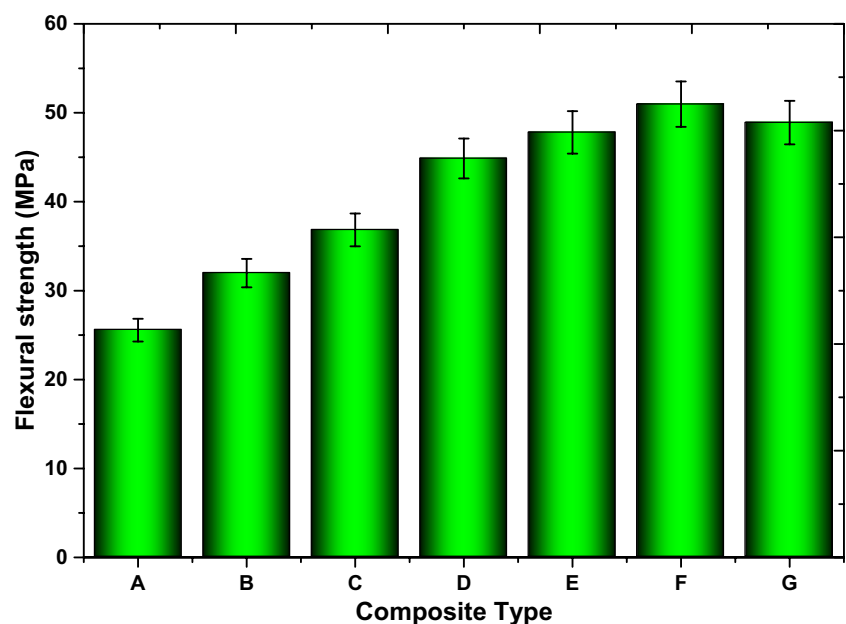


increased from 2.5 wt% to 12.5 wt%. It clearly indicates the presence of nano filler supports the tensile strength. The addition of SiO_2 in excess of the optimum range (≥ 10 wt%) may diminish the impact characteristics; a rise in the SiO_2 proportion may result in greater particle-particle engagement instead of the engagement of the SiO_2 with the strand and matrices. Increased filler materials may result in more micro gaps among the SiO_2 and the substrate; also, SiO_2 agglomeration may decrease its interfacial connection with the matrix, reducing the mechanical characteristics of the laminated materials [38]. This was most likely due to nano- SiO_2 aggregation, as shown in SEM image (Fig. 9d).

3.2.2 Flexural Properties

The bending behaviour of flax fiber-PP composites altered by the incorporation of nano- SiO_2 is shown in Fig. 7. The inclusion of nano- SiO_2 was increase the flexural behaviour of flax fiber-PP materials in the same way that it improved their tensile characteristics. The composites containing 10% nano- SiO_2 had a maximum bending strength of 50.97 MPa, representing a 27% improvement. Flexural performance has improved, which may be attributed to the SiO_2 filler addition's improved matrix and matrix-fiber interface characteristics. As a bonding agent between the filaments and the

Fig. 7 Flexural properties of Flax fiber and different weight proportion of nano SiO_2 based hybrid composites



matrix, fillers are used. Additionally, by adding fillers to the matrix, heat stress at the matrix-fiber interaction is reduced, which enhances interfacial bonding [39]. This increment was exhibited because of the effective load transference from matrix to reinforcement as a result of strong ester, hydrogen, and polyamide linkage formation between flax fiber and PP, along with excellent dispersion of nanostructured SiO₂. It was also expected nano filler must have reduced the gap at the interface of reinforcing flax fiber and matrix. The production of agglomerates may have a harmful effect on filler loading of more than 10% weight%. In actuality, the agglomerates serve as stress concentration zones that might promote the formation and propagation of cracks [40].

3.2.3 Impact Strength

The Charpy unnotched impact quality of flax fiber-based, light-weight composite material is shown in Fig. 8. The impact strength of the hybrid composites steadily rose from 1.12 to 2.89 kJ/m² as the nano-SiO₂ concentration grew from 0 to 12.5 wt%. The improvement in impact strength may be suggest that nano-SiO₂ has greater durability and permeability than flax fibre. As a result, the composites produced by focusing on stress zones needed a greater amount of energy to originate or disseminate a fracture, enhancing the impact resistance of composites. According to the findings, the integration of nanosized SiO₂ impacts the mechanical properties of composite layers due to the excellent distribution of the filler in the substrate produced by mechanical stirring. This results in a strong link between the reinforcement and the resin, allowing for efficient stress transmission among the composite layers [41]. The addition of filler may diminish the available voids, increasing the rigidity of the laminated

materials. Among different weight proportion of nano SiO₂, 10 wt.% of the filler can connect the substrate and the fabric, resulting in greater interaction among the composite laminates. When a load is applied, pressure can be easily transferred from the polymer substrate to the flax fibre, enhancing their impact durability [42].

3.3 Microstructural Analysis

The fractured surfaces on both the plain flax fabric/PP specimen and multiscale hybrids with various SiO₂ weight fractions are shown in Fig. 9. There is poor fibre-matrix interface bonding visible for the tidy mixture (Fig. 9a). The structure of the matrix remnants on the outermost layer of the flax fibres and nano-SiO₂ for the multiscale aggregates (Fig. 9b to d) shows improved additives and matrix adherence relative to the clean hybrid. It is most likely the primary cause of the multiscale composites' superior tensile characteristics compared to those of the clean specimen. The matrix's breakdown surface in Fig. 9a is uniformly smooth and flat, indicating the brittle failure mechanism. The images show that the hybrids devoid of nano-SiO₂ had a higher propensity to develop bigger crystalline and pore spaces, whereas the overall structure was less compacted. Due to the nature of the fracture deflecting process, the framework for the multiscale materials exhibits an erratic and rough fractured surface (Fig. 9b to d) [43]. The SEM observations of the fractured tension sample of the mixture (10 wt%) of SiO₂, flax, and PP are shown in Fig. 9c. Flax fibres are completely coated and saturated with polymers, and SiO₂ and flax fibres are shown to be firmly embedded in the underpinning matrix. No gaps or obvious cavities are visible among the bottom matrices and fibres. The fibres' arrangement within

Fig. 8 Impact properties of Flax fiber and different weight proportion of nano SiO₂ based hybrid composites

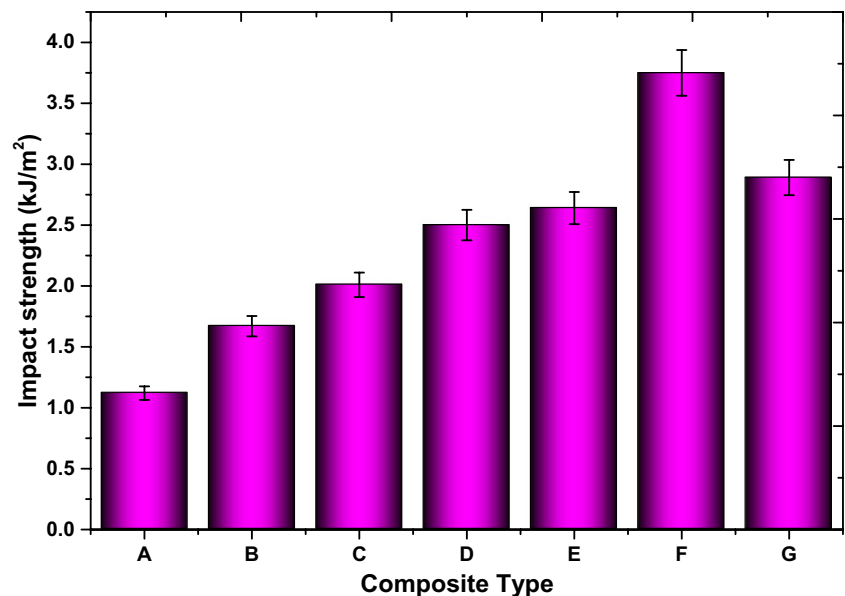
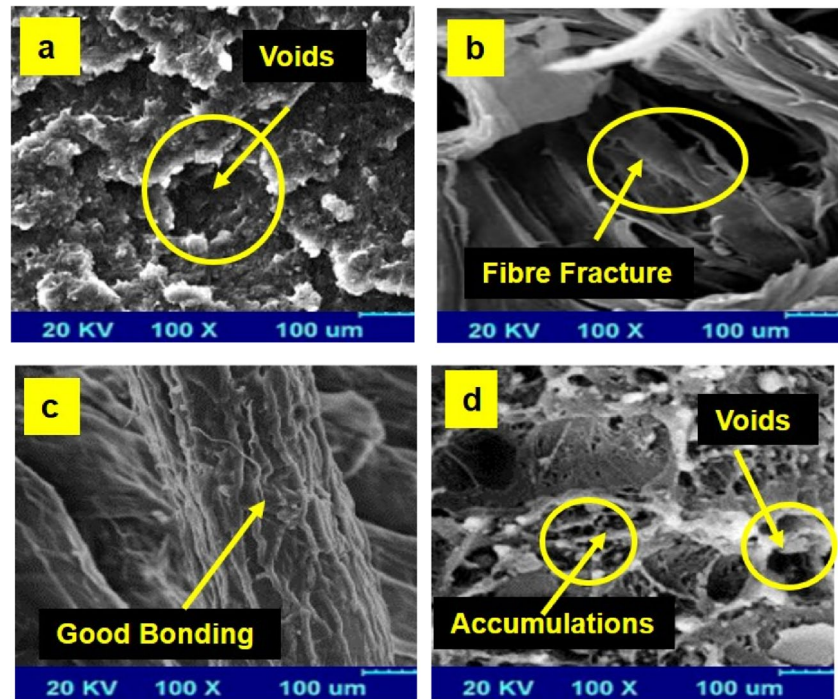


Fig. 9 Microstructural images of **a** pure flax; **b** 2.5 wt% of SiO₂; **c** 10 wt% of SiO₂ and **d** 12.5 wt% of SiO₂/flax based hybrid composites



the structure is important, and they seem evenly dispersed at each of the matrices' four extremes. Effective binding at the intersection of flax fibre, SiO₂, and basic matrices leads to good fiber-polymer interactions. SiO₂ and flax fibre are completely attached to the polymer structure and support the composite. The 12.5 weight% of nanoSiO₂/flax-based hybrid materials is depicted in Fig. 9d. It makes the build-up of filler in the matrix system quite obvious. This causes the rigidity and strength of cracked composites to decrease [44].

3.4 Flammability Characteristics

3.4.1 Impact of Flax Fiber

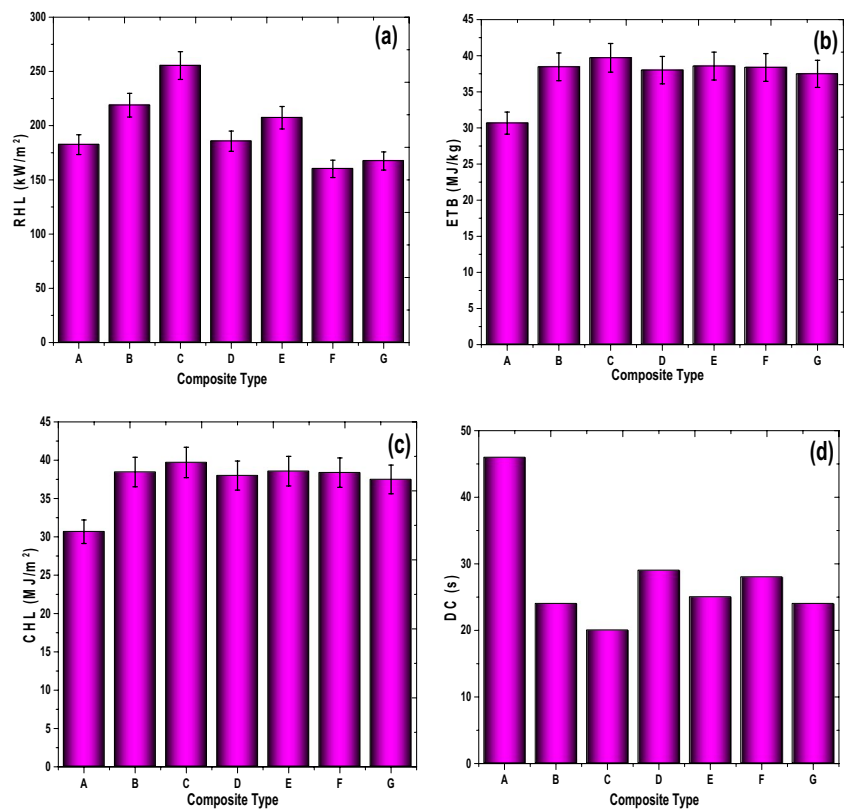
The conical-shaped calorimeter was used to assess the combustibility characteristics of nano-SiO₂-flax fibre-PP-based materials, and the findings of the rate of heat liberation (RHL), efficient thermal burning (ETB), cumulative heat liberation (CHL), weight loss percentage (WLP), peculiar disappearance region (PDR), produced CO, produced CO₂, duration of combustion (DC), and overall emissions emit (OEE) are summarised in Fig. 10. While the flame came into contact with neat PP, it began dripping right away, and the substance's reliability entirely vanished. The inclusion of flax fibre improved this behaviour, and the composite remained intact throughout the trial. In addition, according to the data in Fig. 10, the flax fibre minimised the polymeric WLP by 10.36%. This is most likely due to the flax fibre's lignocellulosic nature. The addition of flax fibre to the polymer increased the RHL by 21.1%, the ETB by 39.21%, and

the CHL by 112.54%, respectively. The PDR was improved, whereas carbon monoxide and carbon dioxide output rose considerably. This could be related to the reality that flax fibre functions as an effective combustible substance, increasing its fire dangers. Tables 4, 5, 6, 7, 8, 9, 10, 11 and 12 explain the calculation of the standard deviation for the cone calorimeter analysis [45, 46].

3.4.2 Impact of Nano Silicon Dioxide

Figure 11a depicts the effect of nano-SiO₂ on the rate of heat liberation of clean PP and flax fibre-based PP-based lightweight materials. The RHL of flax fibre-PP hybrids reduced from 255 to 161 kW/m² when the nano-SiO₂ concentration increased from 5% to 12.5 wt%; that had been nearly 18% less than the lightweight material without nanofillers. Table 13 indicates the summary of cone calorimeter analysis of flax/nanoSiO₂-based hybrid polypropylene composites based on the value of standard deviations. The majority of the reduction in RHL was observed with nano-SiO₂ concentrations up to 10% wt, representing a 26.34% drop. Because of the greater dispersion of SiO₂ particles and stronger interfacial attachment of flax fibre-based PP materials, this study showed that there's an essential impact of SiO₂ packing on fire retardancy. The CHL of flax fibre-PP-based hybrids is depicted in Fig. 11b. An abrupt crest in CHL may be seen at around 110 s. After 170 s, the CHL of flax fibre-PP-based hybrids steadily reduced as the SiO₂ content increased from 0 to 12.5 wt%. The obstacle process may clarify the fire retardancy of silicon dioxide. Throughout the heat transmission process

Fig. 10 Calorimetry results of Flax/SiO₂ based hybrid composites **a** Rate of heat liberation; **b** Efficient thermal burning; **c** Cumulative heat liberation; **d** Duration of combustion; **e** Weight loss percentage; **f** Peculiar disappearance region; **g** Yield of CO; **h** Yield of CO₂; **i** Overall emissions emit

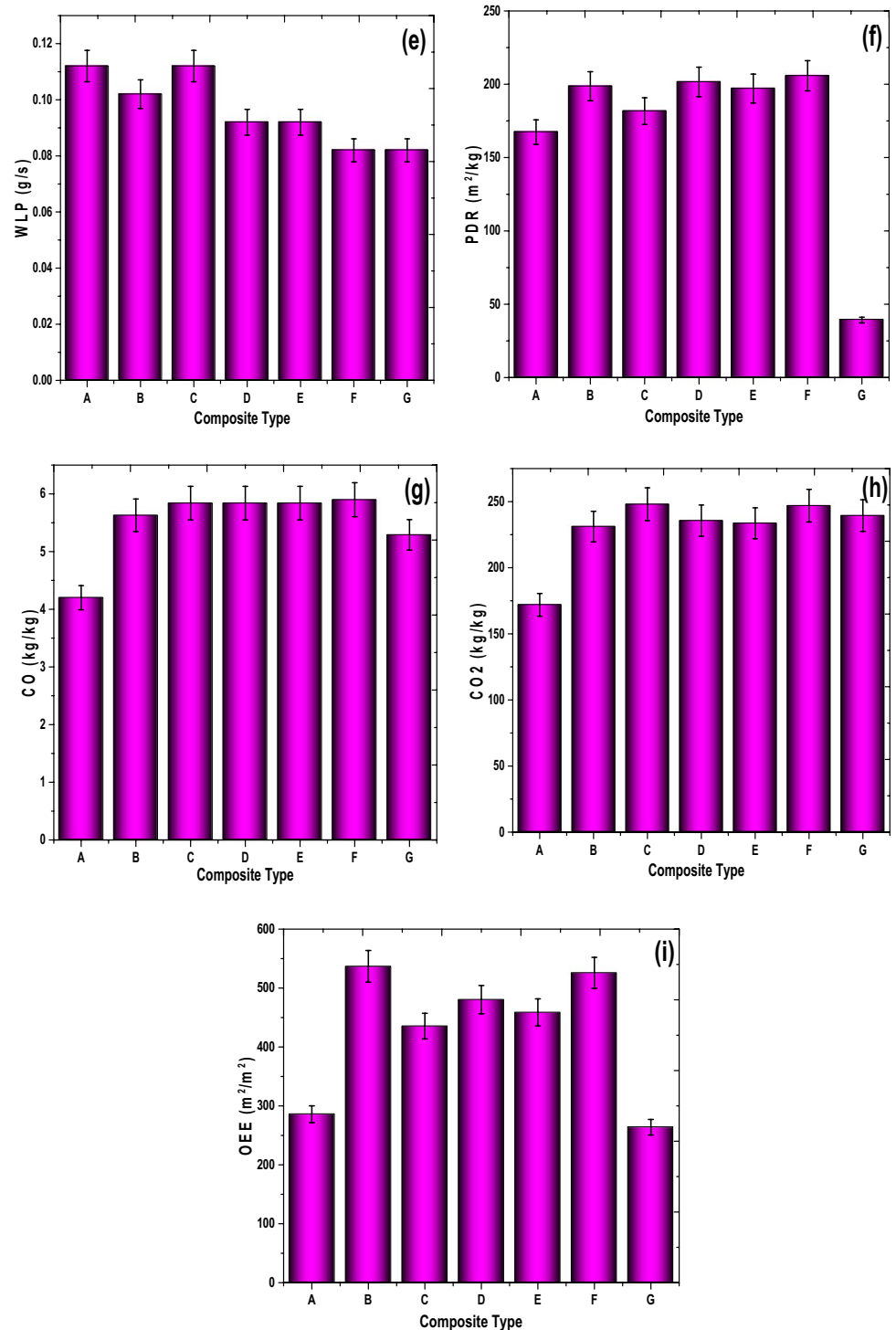


in materials, nano-SiO₂ functions as a radiator and blocks oxygen from igniting a fire by generating a barrier of protection as a covering [26, 47]. Moreover, the incorporation of SiO₂ filler into flax fibre-PP-based hybrids reduced the CHL substantially, whereas the incorporation of SiO₂ filler had no discernible impact on the ETB. This demonstrated that SiO₂ fillers function as streamlined phase fire suppressants. Uncompleted combustion products, including CO and smoke, were also discovered and analysed. On the one hand, it may give a rating of fire dangers; on the other, it might help in the study of fire retardancy systems. The OEE rate of flax fibre-PP hybrids modified by SiO₂ filler is depicted in Fig. 11c. The inclusion of SiO₂ filler lowered the overall amount of smoke produced. Nevertheless, it had no discernible impact on the rate of carbon monoxide and carbon dioxide generation. Because it is more porous and PDR, SiO₂ filler might take in a lot of smoke throughout the process of pyrolysis and burning of materials, reducing overall smoke output [48]. As a result, SiO₂ filler had a significant impact on smoke reduction in polymeric composites. The weight loss of flax fibre-PP hybrids is shown in Fig. 11d, and the inclusion of SiO₂ filler somewhat reduced the weight of the hybrids. The remains of the burned SiO₂-filled flax fibre-PP hybrids displayed firmness with a white surface layer when contrasted with flax fibre-PP hybrids impacted by ammonium polyphosphate in a prior work. The aforementioned intumescent section might provide adequate protection for the composite materials [25, 28, 37].

3.5 Water Absorption Behaviour

The moisture absorbing curves of SiO₂-filled/flax and PP-based hybrids are shown in Fig. 12. As can be seen, all SiO₂-based materials display normal polymer moisture absorbing activity that follows Fick's law [49]. Normally, the inclusion of nanosized SiO₂ reduces the water absorption of flax-based materials as compared to clean PP. This phenomenon is caused by the nanofillers' exceptional barrier characteristics. The inclusion of SiO₂ with an enormous aspect ratio can provide a difficult channel for molecules of water to infiltrate into the aggregates. The maximal water absorption of flax and nano-SiO₂-strengthened materials reduces steadily as the SiO₂ concentration increases. However, the inclusion of flax fibre in a PP system improves liquid intake. This refers to the fact that the flax fibre collected greater amounts of water due to its high amount of cellulose [50, 51]. While contrasted with pure PP as well as PP with flax fibre, the greatest absorption of water in nano SiO₂-filled flax/PP hybrids reduces by 13.25, 15.69, 20.16, 23.63 and 19.52% following the incorporation of 2.5, 5, 7.5, 10 and 12.5 wt% of SiO₂ filler accordingly. The absorption of water diminishes due to the rising amount of filler in all hybrids. This can be attributed to the tortuous impact rising with filler concentration. A number of investigations found that the existence of nanofiller reduced the highest-value water uptake of

Fig. 10 (continued)



polymeric systems. Becker et al. [29] found that various kinds of epoxy frameworks supplemented with multilayer silicon had the lowest water absorption. Zhao and Li [51] examined the liquid absorbed by Al₂O₃/epoxy hybrids in the same way. The fluid absorption of epoxy was reduced with the inclusion of Al₂O₃ tiny particles, according to the findings.

4 Conclusion

In the current research, the natural flax/nanoSiO₂-based hybrid polypropylene materials have been successfully fabricated through the vacuum backing method and found their mechanical, flammability, and water absorption characteristics. The performance of the hybrids

Table 4 Standard deviation of RHL (kW/m²)

Specimen Type	Trail I	Trail II	Trail III	Mean Value	Standard deviations (±)
A	182	181	183	182	1
B	219	219	219	219	0.24
C	255	255	256	255	0.07
D	185	186	189	187	2
E	207	208	207	207	0.32
F	159	161	160	160	0.94
G	167	167	167	167	0.04

Table 5 Standard deviation of ETB (MJ/kg)

Specimen Type	Trail I	Trail II	Trail III	Mean Value	Standard deviations (±)
A	31	30	31	31	0.28
B	38	39	39	38	0.55
C	40	40	39	40	0.29
D	38	39	39	39	0.58
E	38	39	38	39	0.73
F	38	38	39	38	0.40
G	38	37	37	37	0.45

Table 6 Standard deviation of CHL (MJ/m²)

Specimen Type	Trail I	Trail II	Trail III	Mean Value	Standard deviations (±)
A	42	42	41	42	0.58
B	83	84	83	83	0.55
C	78	77	78	78	0.15
D	73	73	73	73	0.11
E	73	73	73	73	0.16
F	77	77	77	77	0.02
G	69	70	69	69	0.60

Table 7 Standard deviation of DC (s)

Specimen Type	Trail I	Trail II	Trail III	Mean Value	Standard deviations (±)
A	46	46	46	46	0
B	24	24	24	24	0
C	20	20	20	20	0
D	29	29	29	29	0
E	25	25	25	25	0
F	28	28	28	28	0
G	24	24	24	24	0

Table 8 Standard deviation of WLP (g/s)

Specimen Type	Trail I	Trail II	Trail III	Mean Value	Standard deviations (±)
A	0.11	0.12	0.11	0.11	0.004
B	0.10	0.10	0.10	0.10	0.001
C	0.12	0.11	0.11	0.11	0.003
D	0.09	0.08	0.09	0.09	0.008
E	0.10	0.09	0.09	0.10	0.004
F	0.08	0.09	0.08	0.08	0.005
G	0.09	0.09	0.08	0.09	0.005

Table 9 Standard deviation of PDR (m²/kg)

Specimen Type	Trail I	Trail II	Trail III	Mean Value	Standard deviations (±)
A	167	168	168	167	0.36
B	199	198	198	199	0.33
C	181	182	182	182	0.73
D	202	202	201	202	0.11
E	197	197	197	197	0.08
F	205	206	206	206	0.62
G	40	39	39	39	0.33

Table 10 Standard deviation of CO (kg/kg)

Specimen Type	Trail I	Trail II	Trail III	Mean Value	Standard deviations (±)
A	4	5	4	4	0.35
B	6	5	5	6	0.42
C	6	6	6	6	0.16
D	6	6	6	6	0.25
E	6	6	6	6	0.22
F	6	6	6	6	0.17
G	5	5	5	5	0.07

Table 11 Standard deviation of CO₂ (kg/kg)

Specimen Type	Trail I	Trail II	Trail III	Mean Value	Standard deviations (±)
A	173	171	172	172	0.99
B	232	231	230	231	0.73
C	248	248	248	248	0.05
D	236	235	236	236	0.55
E	234	233	234	234	0.55
F	248	248	248	247	0.16
G	239	239	240	239	0.53

Table 12 Standard deviation of OEE (m^2/m^2)

Specimen Type	Trail I	Trail II	Trail III	Mean Value	Standard deviations (\pm)
A	286	285	286	286	0.74
B	537	537	536	537	0.74
C	436	435	435	435	0.53
D	481	480	480	480	0.60
E	459	459	458	459	0.30
F	526	526	526	526	0.39
G	264	264	264	264	0.10

has significantly improved as a result of the addition of nanoSiO₂ particles to the polypropylene matrix. Based on the obtained results, the following conclusion was drawn:

- First off, the reinforcement offered by the flax fibres and 10% of nanoSiO₂ particles significantly improved the mechanical characteristics of the hybrids. The aforementioned substances were added to the hybrids to increase their tensile strength, flexural strength, and impact resistance, which made them suitable for a variety of load-bearing uses. While aggregation was seen in hybrids with 12.5 wt% nano SiO₂, according to the

SEM analysis on the fractured surface of the materials. A comparable impact was shown in the hybrids of natural fibres and nanoparticles, which led to a simultaneous enhancement in mechanical properties.

- In addition, flammability experiments showed that the polypropylene composites' flammability was significantly decreased by the inclusion of natural flax fibres and nanoSiO₂ particles. Although exhibiting no noticeable impact on ETB, the inclusion of nano-SiO₂ can reduce the RHL and OEE of flax fibre-PP hybrids. With 10 weight% nano-SiO₂, RHL decreased primarily. Cone calorimetry measurement research revealed that the nano-SiO₂ on flax fibre/PP composites served as a condensed-state flame retardant throughout burning. This is due to the natural flame-retardant qualities of flax fibres as well as the synergistic action of nanoSiO₂, which functions as an external barrier and a heat sink, impeding the expansion of fires and containing them.
- Furthermore, the moisture-absorbing qualities of the hybrids were examined, and it was discovered that the inclusion of flax fibres and nanoSiO₂ particles significantly decreased the ability of the polypropylene matrix to absorb moisture. The amount of water consumed in flax/PP hybrids decreases by 13.25, 15.69, 20.16, 23.63 and 19.52% as compared to pure PP and PP with flax fibre

Fig. 11 Influences of nano silicon dioxide addition to Flax/PP composites by cone calorimetry analysis **a** Rate of heat liberation, **b** Cumulative heat liberation, **c** Overall emissions emit, and **d** Weight loss proportions

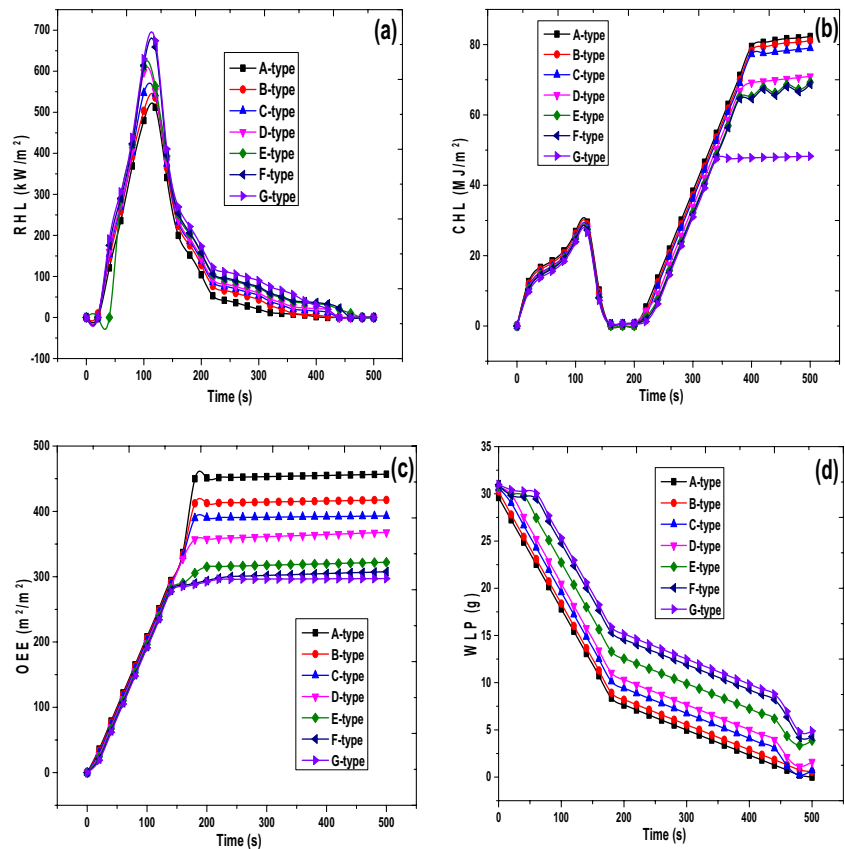
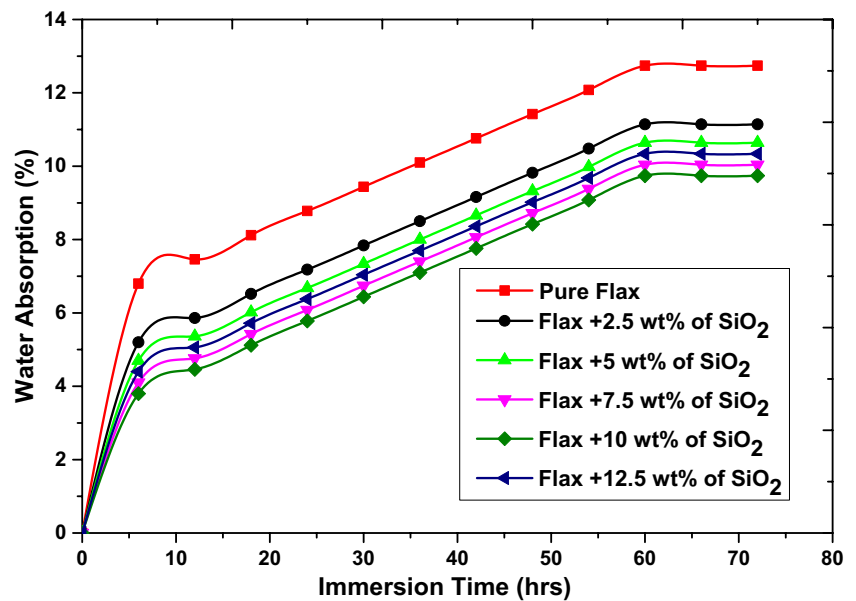


Table 13 Summary of cone calorimeter analysis of Flax/NanoSiO₂ Based hybrid Polypropylene Composites

Specimen Type	RHL (kW/m ²)	ETB (MJ/kg)	CHL (MJ/m ²)	DC (s)	WLP (g/s)	PDR (m ² /kg)	CO (kg/kg)	CO ₂ (kg/kg)	OEE (m ² /m ²)
A	182	31	42	46	0.11	167	4	172	286
B	219	38	83	24	0.10	199	6	231	537
C	255	40	78	20	0.11	182	6	248	435
D	187	39	73	29	0.09	202	6	236	480
E	207	39	73	25	0.10	197	6	234	459
F	160	38	77	28	0.08	206	6	247	526
G	167	37	69	24	0.09	39	5	239	264

Fig. 12 Water absorption behaviour of Flax/PP/Different weight proportion of nano SiO₂ based hybrid composites

with the addition of 2.5, 5, 7.5, 10 and 12.5 wt% of SiO₂ filler, respectively. Decreased water absorption avoids dimensional instability, deterioration, and long-term loss of mechanical properties, which is critical for applications in humid conditions.

Overall, experimental findings show that natural flax and nanoSiO₂-based hybrid polypropylene nanocomposite have the potential to be used as materials that are moisture-resistant, flame-resistant, and ecologically benign. Such composites have a lot of potential in sectors like automobiles, building, and wrapping, where there is a need for renewable and high-performance materials. Even more sophisticated composites with specialised qualities might be created as a result of more research and development in this field, paving the way for a future that is more environmentally friendly and sustainable.

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Data Availability The data used to support the findings of this study are included in the article. Should further data or information be required, these are available from the corresponding author upon request.

Declarations

Ethics Approval Not applicable.

Consent to Participate Not applicable.

Consent for Publication Yes. All permission granted.

Competing Interests The authors declare no competing interests.

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