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RESEARCH

Exploring Mechanical and Flammability Traits in Hybrid Composites of Crown Flower/Nano SiO_2/$4ZnO{\cdot}B_2O_3{\cdot}H_2O$ under Cryogenic Conditions: an Experimental Study

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Abstract

One of the trendiest areas in the world of materials research is multifunctional polymer nanocomposites. The long-term behaviour of fibre-reinforced polymer composites exposed to cryogenic environments is discussed in this article in light of current advancements. The composites were created using the standard hand layup procedure in order to achieve the aforementioned goals. Crown flower fibre (CF) serves as a reinforcement, while 3 wt.% of nano-SiO_2 and 3 wt.% of zinc borates ($4ZnO{\cdot}B_2O_3{\cdot}H_2O$) serve as fillers, and the LY566 type of epoxy resin with the HY 951 type of hardener were considered as a matrix system. To complete the mentioned objectives, three types of composites were fabricated: pure epoxy-based CF laminate, CF/3 wt.% of nano SiO_2 hybrid, and CF/3 wt.% of nano $4ZnO{\cdot}B_2O_3{\cdot}H_2O$-based hybrid laminates. Analysis of the mechanical behaviour of composite materials that had been created was done under both normal and cryogenic circumstances at two distinct temperatures (−60 and 30 °C). To investigate the flammability properties of the hybrid composites, tests with horizontal and vertical flame retardants were conducted. X-ray diffraction and Fourier transform infrared spectroscopy studies were used to validate the nanofiller's existence. The manufactured CF/3 wt.% of SiO_2-based hybrid composites have good mechanical capabilities in a cryogenic environment of −60 °C, according to the results. This is because the 3 wt% SiO_2-loaded samples are more resistant to deformation and can take more force before breaking at the impact zone because they have a larger surface area per volume fraction and better adhesion. In terms of flammability characteristics, the hybrid CF/ 3 wt.% of $4ZnO.B_2O_3.H_2O$ (zinc borate) exhibits better results (burning rate of 7.14 mm/min). The burning rate was reduced as a result of the use of nanofillers. This is due to zinc borate's promising flame-retardant qualities, which prevent flame spread and aid in flame extinguishment.

Keywords Crown flower · Nano SiO_2 · Nano $4ZnOB_2O_3H_2O$ · Tensile strength · Cryogenic treatment · Flame Retardancy

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1 Introduction

When two or more material elements with various qualities are combined, the final product frequently produces a lightweight framework with enhanced rigidity and characteristics that are specifically designed for particular uses where weight reduction, improved strength, and lower energy consumption are essential [1, 2]. It is widely understood that the main two components of the material are the matrices and the fibre or filler; the matrix acts as a binding agent that holds the fibres together to produce the composite material. Composites gain stiffness from the matrix, which helps reinforce their strength to handle extreme tensile stresses. When stress is applied to a composite system, the fibres frequently act as load-transfer mechanisms that cause stresses to be transferred from one fibre to a different one, which are then cemented collectively in a matrix [3, 4]. Degradation of composite substances may also be attributed to inadequate matrices at the fibrous junction and the position of the fibres [5]. It has been demonstrated that adding either organic or inorganic additives to composites is an efficient technique to increase the mechanical rigidity and strength of substances, in addition to offering answers to the problems with composite failures stated previously [6]. Among the plant-based fibres examined were jute, hemp, sisal, Crown flower fibre, and Kenaf. Crown flower fibre, sometimes referred to as gigantic milkweed or crown blossom, is an organic material made from plants that has several uses [7–9]. A perennial species known as Crown flower, it is indigenous to tropical and subtropical regions of Africa, Asia, and Oceania. Traditional applications of the fibre obtained from the Crown flower plant include the production of chains, cables, netting, and fabrics. It has special qualities that make it suitable for a variety of uses [10, 11]. The potential of Crown flower fibre in contemporary usage, such as ecological fabrics, green packaging, and agricultural commodities, has recently attracted increased study. Investigators and developers are looking at ways to get beyond the difficulties involved in extracting and processing it, hoping to use its special qualities for a variety of sectors while taking biodiversity and the regional economy into account [12, 13].

Long-term contact with cold temperatures causes considerable changes in the mechanical characteristics of composite substances under stress. At −196 °C in comparison with that at 23 °C, the durability of glass fibre/epoxy laminated materials rose by 30 to 40%, while the toughness of carbon fibre/epoxy laminated materials improved by 15 to 20% [14]. The rigidity and mechanical durability of the polymeric matrix hybrid are increased at temperatures below freezing by the addition of micro and nanofillers. Additionally, the strength and rigidity of polymers are impacted by their void percentage. Owing to the in-plane thermal stresses that are created at cryogenic conditions, the impact on the properties of composite elements varies. Microcracks appear in the framework due to the larger variance in the thermal contraction of the polymer and carbon/epoxy fibre composite [15, 16]. At lower temperatures, the breaks make the interfaces that connect the carbon fibre substrate weaker. Following 10 days of treatment at −55 °C, the mechanical characteristics of glass fibre-strengthened materials, including bending strength, compression strength, and tensile force, significantly improved. The dimensional strength and stability properties of the glass epoxy filled materials were improved by the inclusion of powdered graphene up to 7.5 weight percent filler [17, 18]. The matrix's fibre interaction and consistent distribution of filler increase the composite's tension strength, according to the association between morphology and tension strength. In comparison to the pristine materials, the pre-stressed elastic polymer matrix materials exhibit superior retention of energy at cryogenic conditions [19]. The matrix formed by the fibre-separating process is an energy absorbing process.

Materials used in many different sectors, particularly those requiring manufacturing, automobiles, gadgets, and fabrics, must have flame retardant qualities. Because of their potential for environmental advantages and distinctive features, natural composites, a type of material created by fusing natural fibres with a matrix substance, have attracted interest. Such composites may be created by combining compostable matrix like starch, soy, or polylactic acid with plant-based threads, including jute, flax, hemp, and sisal [20]. Whenever confronted with a source of heat or a flame that is open, a substance's ability to withstand or delay the propagation of flames is referred to as its flame-retardant properties. Natural composites have built-in benefits in terms of their flame-retardant qualities. When using a polymer composite substance in a real time application, it is important to evaluate and investigate its combustibility performance and resistance to water [21, 22]. All polymer compounds ignite quickly and produce a lot of heat, fire, and smoke when subjected to fire since they are fundamentally made up of hydrocarbon strands. Because natural fibres possess better burning qualities than artificial fibres produced by the natural world, combustibility tests are specifically carried out for polymers supplemented with organic fibres. When fly ash is added, the combustibility of weaved jute fibre-strengthened fly ash-loaded polymeric composites is dramatically reduced, according to research by Suriani et al. [23]. A combined material with an acceptable combustion rate of 10.2 mm min1 in the horizontal UL-94 test is produced by including 5 weight percent of jute fibre, 15 weight percent of

fly ash, and 10 h of NaOH processing. Reaction and additive flame retardants are usually added to natural fibres to increase their flame resistance. Diols and polyols, including phosphorous, were often added to fibres made from nature as reactive retardants [24]. Nevertheless, the single use of reacting flame retardant was unable to achieve the desired flame suppression at a cheap cost due to the inadequate flame-retardant efficacy as well as the expense. The most common approach to improving the fire resistance of natural fibres was to blast flame retardants into polymers. The following inorganic materials, like hexaphenoxycyclotriphosphazene, ammonium polyphosphate, expandable graphite, dimethyl methyl phosphate (DMMP), triethyl phosphate, red phosphorus, nitrogen phosphorus, melamine derivative, nano SiO_2, and other flame retardants, have been added to enhance the combustibility of organic fibre [25].

According to earlier studies in the field of polymeric materials, hybrids made of an artificial fibre and polymers that were subjected to temperatures below freezing showed improved mechanical characteristics compared with materials made at room temperature. Yet, only a small amount of research has been done on how bidirectional natural fibre composites behave below zero degrees Celsius. In this section, we examine the potential for adding fillers such as nanoSiO_2 and $4ZnOB_2O_3H_2O$ to strengthen epoxy-based materials reinforced with Crown flower fibre (CF). To have a better understanding of how the hybrid CF/epoxy composites behave at ambient temperature and below zero degrees, the water absorption, flammable, and mechanical features are quantitatively studied.

2 Experimentations

2.1 Materials

Araldite LY 556, an epoxy matrix, was used as the manufacturing material for the composites. Improved matrix effectiveness is achieved when 10% by weight of Aradur HY 951 hardener is incorporated into the resin. Nano SiO_2 (231.533 g/mol of molecular weight with a 25 nm size) and $4ZnOB_2O_3H_2O$ (313.7584 g/mol of molecular weight with a 25 nm size) were purchased from Deekshitha Chemicals in Bangalore, Karnataka, India, and both materials were 99% pure. Crown flower fibre, which is bidirectional and 520 GSM, is used as reinforcement for hybrid fabrication and could be purchased at the Rithu fibre industry in Salam, Tamil Nadu, India.

Zinc borate is commonly utilized due to its effective flame-retardant properties across a range of materials. Its selection can be attributed to various factors, such as its potent fire-inhibiting characteristics, including:

- **High decomposition temperature:** At a comparatively high temperature, zinc borate breaks down, releasing water vapour and covering the material's surface in a layer of protection. This may aid in stopping the combustion process or reducing its speed.
- **Synergistic effects:** In order to get beneficial interactions, zinc borate is occasionally used in conjunction with other flame retardants. This implies that combining several flame retardants can increase their overall efficacy [26].
- **Low toxicity:** Zinc borate is thought to be comparatively less harmful than certain other flame retardants, which makes it a safer choice in some applications, particularly in goods that come into contact with people or animals.
- **Compatibility with polymers:** Because zinc borate frequently works well with a wide range of polymers, it may be used in a number of materials, including textiles, rubber, and plastics.
- **Smoke suppression:** Zinc borate has flame-retardant qualities as well as the ability to help reduce emissions of smoke during combustion, which is advantageous for applications involving fire safety [27, 28].

Simultaneously, because of its fire resistance, silicon dioxide (SiO_2) is frequently used as a filler or flame retardant in a variety of materials. It may function as a physical barrier and has a high melting point, which lowers the flammability of materials. Furthermore, when exposed to flames, silicon dioxide may absorb heat and emit water vapour, which helps to extinguish fires [29]. For the reasons outlined above, zinc borate and silicon dioxide were chosen as the flame retardants for the current study.

2.2 Pre-treatment of CF

The CF fibres were first carefully cleansed and prepped to get rid of any oils and pollutants that could be on the exterior. The cellulose fibres are then submerged for 24 h at 60 °C in a concentrated (5 weight percent) sodium hydroxide solution. The fibres expand and undergo morphological modifications as a result of this approach. The fibres are gently washed with water to eliminate any extra sodium hydroxide once the appropriate amount of time has passed. Any leftover alkali on the fibre surfaces is neutralised with an acid solution (diluted in acetic acid) in order to stop further reactivity with the fibres. The CF fibres are then completely dried to return them to their usual moisture content.

Table 1 Different types of fabricated composites

Sl.No	Specimen type	Composition
1	Specimen 1	Pure CF based composites
2	Specimen 2	CF/3wt.% of Nano SiO_2
3	Specimen 3	CF/ 3 wt.% of Nano $4ZnOB_2O_3H_2O$

2.3 Composite Fabrication

To suit the steel mould, a 300 mm × 300 mm piece of Wowen CF fibre mat was fashioned. SiO_2 and $4ZnOB_2O_3H_2O$ nanomaterials have been dried in a furnace at 80 °C. Table 1 shows how the different types of epoxy composites are made, along with their composition. In order to prepare the epoxy/nano composite, epoxy resin was combined with 3 weight percent SiO_2 and $4ZnOB_2O_3H_2O$ using a stirring machine until thoroughly homogenised. The combination of the curing agent and epoxy was mixed in at a ratio of 1:10 each. The ingredients were well mixed before being evenly poured into the mould in order to begin the process of curing. The CF fibre mats were positioned on top of a thin coating of epoxy resin that was in the moulds for the creation of epoxy/CF/SiO_2//$4ZnOB_2O_3H_2O$ blended composites, and the additional layer of resin was subsequently placed on top of the fibre mats. When three distinct layers had been achieved, the procedure was continued. Before undergoing the procedure of curing, the metallic sheets had been placed on top of the mixture and tightly clamped. Pre-curing was done at 70 °C for two hours, and post-curing took place at 110 °C in a standard oven. The plastic composites were removed from the mould following the curing process and allowed to cool at ambient temperature before being labelled. Figure 1 shows the complete graphical representation of composite fabrication process.

2.4 Cryogenic Treatments

When polymeric components were made, they underwent cryogenic processing. The cryogenic operation was completed in a controlled, programmed-freeze condition. For the static examination, the combined samples are placed in liquid nitrogen for five hours until the temperature reaches −60 °C. The thermocouple is used to gauge the internal temperature of the laminate plates.

2.5 Materials Characterization

2.5.1 XRD and FTIR Analysis

The nano SiO_2 and $4ZnOB_2O_3H_2O$ incorporated hybrid nano composite samples were characterized by XRD for the crystal structure, average particle size and the concentration of impurity compounds present. Rigaku Rad B Powder X-ray diffractometer was used for X-ray diffraction patterns of these samples. The 2Θ values were taken from 10 to 70

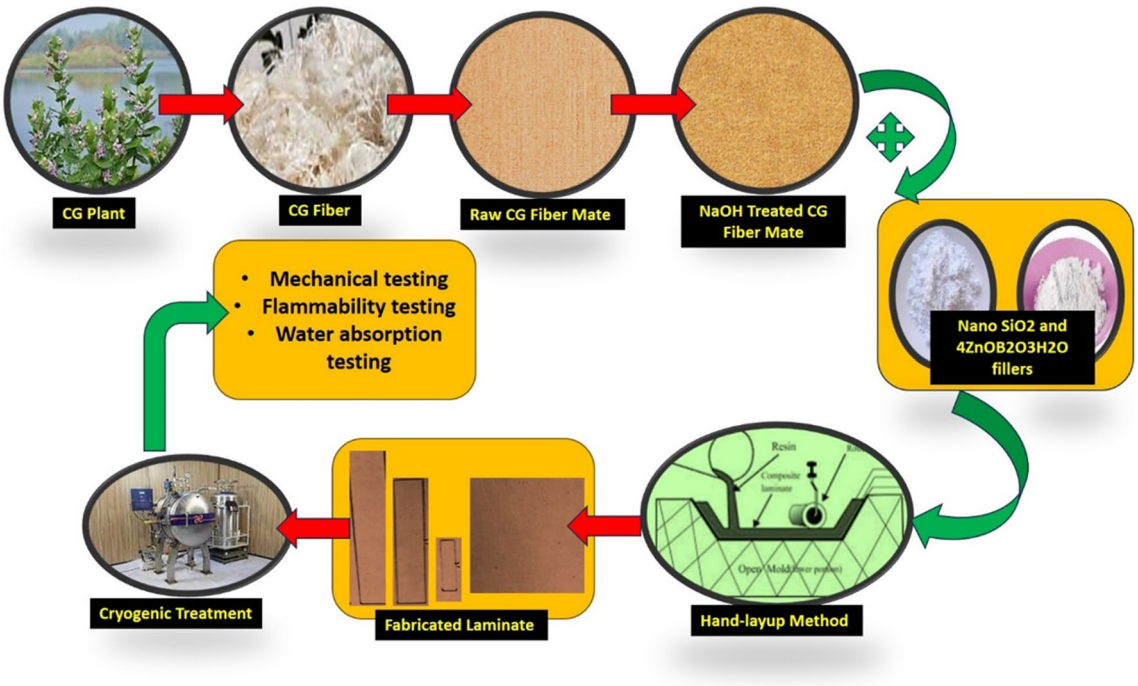

Fig. 1 Graphical representation of the fabrication process of CF/SiO_2/$4ZnOB_2O_3H_2O$ based hybrid composite

with a step size of 0.048 using Cu Ka radiation (α value of 2.2897 A˚). The dried samples were dusted on to plates with low background. Fourier transform infrared (FT-IR) spectroscopy of the samples as powder-pressed KBr pellets were examined in the wave number range from 4000 to 500 cm − 1 using a PerkinElmer 2000 spectrophotometer.

2.5.2 Mechanical Characterization

The CF/epoxy hybrid laminate is put through three-point bending and tensile testing in accordance with ASTM D790 (127 × 12.5 × 3 mm) and ASTM D3039 (250 × 25 × 3 mm), respectively. Shimadzu UTM is used to do mechanical evaluations for tensile force and bending strength. A measurement of the modulus is provided by the degree of distortion, rigidity, and flexibility of the substrate. Calibration is done in a variety of environments, including temperatures below freezing −60°, −30 °C, and the surrounding temperature. The compression measurement is done in accordance with ASTM D3410 (150 × 25 × 3 mm). Compression examination is highly difficult since the specimens are susceptible to collapsing while in a compressed state and the force of compression is applied straight to their ends. According to ASTM D2344 (18 × 6 × 3 mm), interlaminar shear strength (ILSS) testing is a three-point flexural test that encourages the evaluation of material integrity via interface. During the debonding process, the CF/epoxy hybridization composite's microstructural stability is determined by the ILSS.

2.5.3 Flammability Testing

Because of their great flammability, fibres made from nature pose a serious threat to both systems and buildings. As a result, it is crucial to evaluate the flammability traits of the laminated materials that are manufactured. According to ASTM D635 and ASTM D3801 requirements, the manufactured specimen's horizontal and vertical combustible properties were investigated. Cantilevered samples were made, and their horizontal and vertical combustibility have been determined by constantly exposing them to a spirit burner for roughly thirty and ten seconds, respectively. The heating element needs to be moved farther from the sample after 30 s, or when the flame reaches 25 mm. The combustion procedure has to be restarted, and the present moment, marked by t1, has to be kept track of till the flame reaches the 100 mm mark or the process of combustion is finished, whatever occurs first. The expression that follows (1) was used to determine the specimens' combustion percentage.

$$Burning\ Rate = \frac{L_r}{T_r} \times 60 \tag{1}$$

where, Lr is the length of sample burnt (mm) and Tr is the sample burning time (s), respectively.

2.5.4 Water Absorption and Swelling Behaviour

The hybrid materials are tested for water absorption in accordance with ASTM D2344-84. The mass of every sample was first given in a dry state. The CF/epoxy hybrid samples are subsequently immersed in water for varying lengths of time (60 h) to enable uptake at ambient temperature. After the specimen has been submerged in water, any remaining water is wiped out with a piece of tissue. The specimens are weighed using a computerised balance with a precision of 0.001 mg. The samples are weighed repeatedly until saturation limits are reached. The variations in weight between dry and wet circumstances are due to water uptake. The empty and loaded (SiO_2 and $4ZnOB_2O3H_2O$) samples' absorbing water performance was calculated using the subsequent formulae 2.

$$Water\ absorption = \frac{W_2 - W_1}{W_1} \times 100 \tag{2}$$

where W_2 is the weight of the sample after immersion, W_1 is the weight of the sample before immersion.

When subjected to moisture, natural fibre-strengthened composites may retain a certain quantity of water as well as a certain moisture concentration. As a result, the ASTM D570 swelling experiment was conducted. The proportion of swelling seen on the hybrid nanocomposite samples may be determined by comparing the dimensions of the material sample prior to and following the absorption test for water. The aforementioned formula was used to quantify thickness swelling during the course of one day, and specimens were submerged in distilled water for 60 h at ambient temperatures.

3 Result and Discussions

3.1 Characterization of Nanocomposite

3.1.1 X-Ray Diffraction Analysis

Figures 2a and b depict normal diffraction trends from an X-Ray Diffraction (XRD) study of a powdered substance. Through applying the Debye-Scherer formula to these charts, we can determine the sizes of both the SiO_2 and $4ZnOB_2O_3H_2O$ nanoparticles or crystallites, as shown below (3):

$$D = \frac{K\alpha}{\beta Cos\theta} \tag{3}$$

wherein D is the mean crystal dimensions, K is an independent variable of crystal form (0.9), α is the X-ray wavelengths

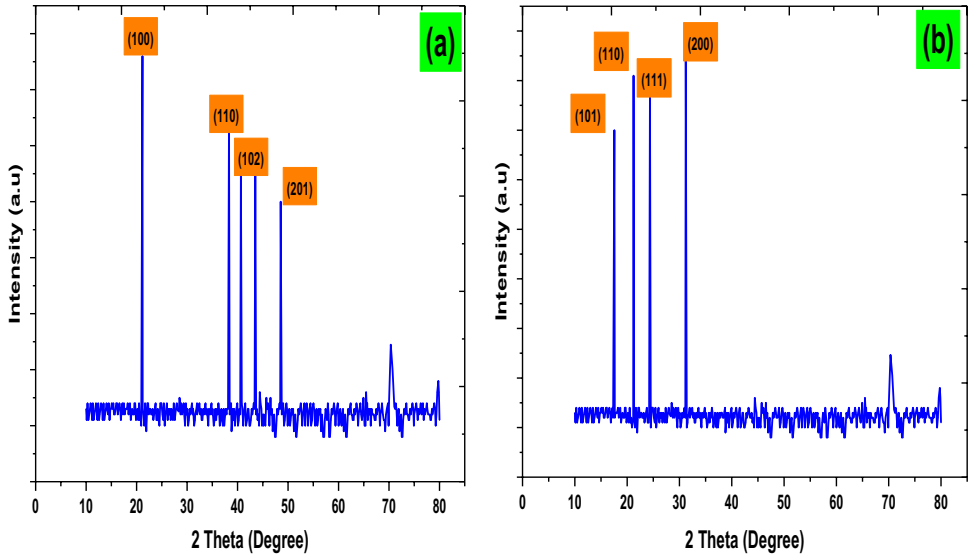

Fig. 2 XRD pattern of (**a**) CF/SiO_2; (**b**) CF/4ZnOB$_2$O$_3$H$_2$O based hybrid composites

(in this example, for Cu-K radiation), ß is a full width at half maximum, and Θ is the Bragg's inclination. We are able to calculate the dimensions of the fragments using the formula provided above. We can make certain that the substance that was established is SiO_2 (Quartz) nanoparticles by contrasting our gathered XRD spectrum with the JCPDS Card No.850335 for SiO_2, as the spikes show the development of particles with dimension in the nm range and reflections via (100), (110), (102), (111), and (201) planes, at 2Θ values of 21.16, 38.30°, 40.66°, 43.51°, and 48.54. The hexagonal crystalline arrangement of the substance that was generated and its fundamental lattice, with the parameters of the lattice a = b = 4.203 and c = 5.210, are both evident from the XRD pattern [30].

The XRD structures of the nano zinc borate are displayed in Fig. 2b. Figure 2b displays a few different peak shapes of pure zinc borate, indicating that the fragments were crystalline. The results from the diffraction information and JCPDS File No. 21–1473 were in reasonable agreement, and every one of the peak values for diffraction was fairly comparable with those of pure 4ZnOB$_2$O$_3$H$_2$O. No recognisable impurity spikes from the other unprocessed chemicals were seen. As a consequence, the findings demonstrated that the product has a monoclinic crystal state (4ZnOB$_2$O3H$_2$O). The production of particulate has dimensions in the nm range as well as reflecting from surfaces at 2Θ values of 17.56, 21.23, 24.36, 28.63, and 31.20º from (101), (110), (111), (202), and (200) [31].

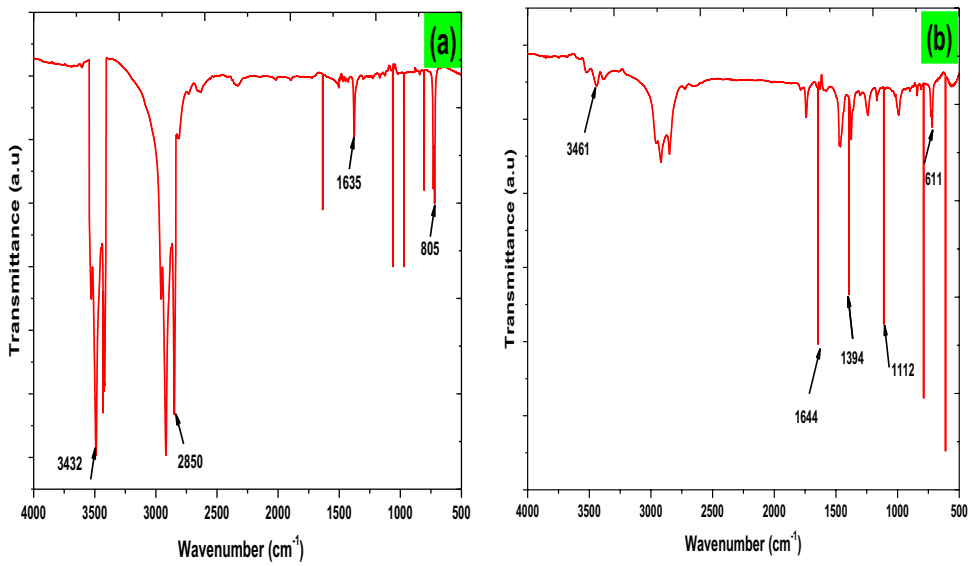

Fig. 3 FTIR analysis of (**a**) CF/SiO_2; (**b**) CF/4ZnOB$_2$O$_3$H$_2$O based hybrid composites

3.1.2 FTIR Analysis

The FTIR spectrum for the resultant silicon dioxide nanoparticle is shown in Fig. 3a. The Si–O bending vibrational group of about 805 cm^{-1} represents oxygen moving at a right angle to the Si–Si bands in the Si–O-Si planes. The deformation motion of Si–OH is shown by its absorption at 971 cm^{-1}. The Si–O-Si band's asymmetrical vibration of stretching occurs in the region of approximately 2850 cm^{-1}, and the atom of oxygen that bridges the gap travels perpendicular to the Si–Si lines in a different orientation from its Si neighbours. Si–OH vibrations that stretch with hydrogen bonds are shown by the band at 3432 cm^{-1}. The silicon dioxide nanoparticles are extremely hygroscopic, according to FTIR measurements. Another peak at 1635 cm^{-1} in the IR spectra has also been found and is linked to Si-H_2O flexing [32].

The infrared spectra of nanometric zinc borate nanoparticles (4000–500 cm^{-1}) are shown in Fig. 3b. The subsequent absorption rates can be seen in the IR spectrum of a pure zinc borate specimen. O–H is stretched in the region of the band at 3461 cm^{-1}. The H–O-H bend mode, that has been assigned to the group at 1644 cm^{-1} reveals a substance, including crystalline water. The asymmetrical bending of B (3)-O may be what is causing the band at 1394 cm^{-1}. The deformation of B (4)-O is identified as the source of the bands at 1112 and 789 cm^{-1}, respectively. The triborate anion's symmetrical pulsed oscillation has been assigned to the frequency range of 611 cm^{-1}, demonstrating that the manufactured specimen includes zinc borate [33].

3.2 Mechanical Properties

3.2.1 Tensile Strength

A material's tensile strength, a critical mechanical characteristic, determines how well it can endure a force of stretching before fracturing or significantly deforming. Tensile strength describes the extent to which an organic fibre or nanofiller-based hybrid can withstand being torn away across its length when exposed to an imposed tension force. Figure 4 shows the tensile strength of hybrid composites. The tension findings show that the inclination of a curve rises as temperatures drop. The resulting values of both strength and stiffness also heavily depend on how fragile the fibres and matrix are. The shrinkage of the matrices is primarily responsible for the greater tension strength at a lower temperature [34]. At low temperatures, the matrix's structure becomes less flexible but more durable and rigid. The framework encounters residual stresses because the CF fibres have clearly lower or low coefficients of thermal contraction (CTE). When external loads are applied to samples at below-freezing temperatures, residual stresses are generated, placing stress on the

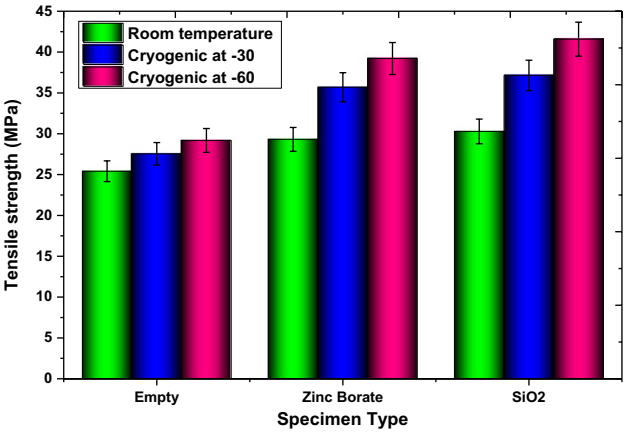

Fig. 4 Tensile strength of CF/nano filler-based hybrid composites under different temperature conditions

mixtures. The minimum energy needed for fracturing the connection among a matrix and fibre raises following the sample's hardening following being subjected to liquid nitrogen and thus raises the energy limit for matrices to collapse owing to the beginning, transmission, and tension breakdown of microcracks at lower temperatures [35].

The stress–strain performance of the hybrid sample shows that after the CF/epoxy hybrid materials were exposed to liquid nitrogen, the glass transition temperature (Tg) increased, increasing the tensile strength. The epoxy's plasticity regulates the mixed materials' nonlinear performance. Tensile strength increases by 15.87% for the empty specimen at -30 °C, 9.23% for the 3-weight percent $4ZnOB_2O_3H_2O$ filled samples, and 13.65% for the 3-weight percent SiO_2 filled samples, as illustrated in Fig. 4. The tensile modulus (TM) of CF/epoxy blended composites follows a similar pattern to the stress–strain response of the various layered composites [36]. According to the outcomes, the introduction of nanofillers can have a number of advantages due to their tiny dimensions and large surface area. It can strengthen the link between the structure of the material and the filaments, thereby decreasing the likelihood of cracks spreading and increasing the material's resilience to distortion. Additionally, by serving as stress-transferring connections, they help distribute the stress more uniformly throughout the substance [37].

3.2.2 Flexural Strength

Flexural strength, also known as bending strength or modulus of rupture, is a mechanical property that measures the maximum amount of bending stress a material can withstand before it breaks or fails. In the context of natural fibre and nanofiller-based composites, flexural strength refers to the ability of the composite material to resist deformation and

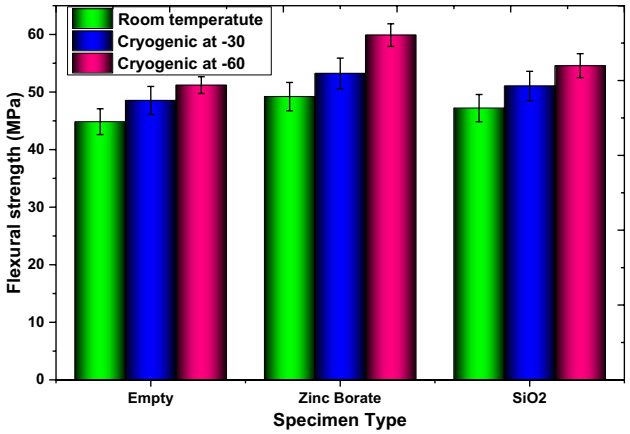

Fig. 5 Flexural strength of CF/nano filler-based hybrid composites under different temperature conditions

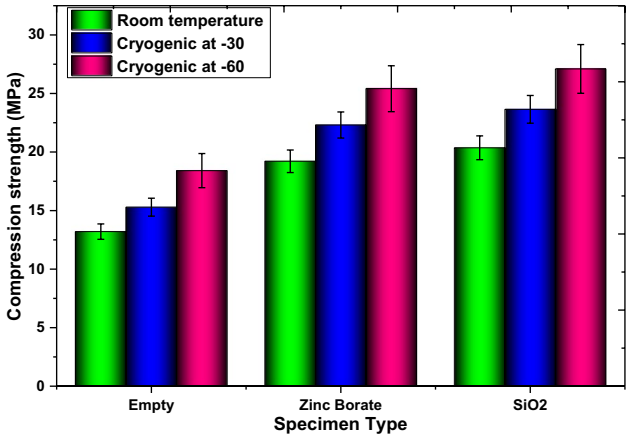

Fig. 6 Compressive strength of CF/nano filler-based hybrid composites under different temperature conditions

fracture when subjected to a bending force. The flexural test is carried out to quantify the effect of temperature on flexural strength [38]. The hybrid specimens exposed to subzero temperatures develop higher flexure strengths compared to the ones tested at room temperature, as shown in Fig. 5. Accordingly, the 3 wt.% $4ZnOB_2O_3H_2O$ loaded hybrids show the greatest improvement in bending strength, having an improvement of 16.32% at −60 °C compared to the ambient temperature, while the lowest change is shown for the 3 wt.% SiO_2 filled composite samples, having a gain of 14.02% at −60 °C. For the empty specimens, the increase in flexural rigidity at −60 °C above the ambient temperature is 12.20%. The figures that show the usefulness of the materials at both temperatures show that the bending strength of CF/epoxy hybrid materials at freezing temperatures is higher than at ambient temperatures. As its flexural modulus drops, the substance becomes more ductile and may withstand significant strain before failing. A highly resilient fibre must break in bending, but the outcomes of tensile tests reveal the breakage of the weakest fibre. This indicates that the flexural strength is greater than the tensile strength [39]. Figure 5 shows the flexural strength of hybrid composites.

3.2.3 Compression Strength

The compression participants, which are components of the structural elements, are either packed in straight compression modes or in conjunction with flexure. The longitudinal rigidity of each component correlates to the cross-section dimension of the entire structure. So, within certain bounds, the cross-sectional shape may be changed to change the rigidity. Low-compressive-strength composites have a restricted range of applications. Figure 6 illustrates that regardless of whether the specimens are full or empty, the compression strength is greater at below-freezing temperatures compared to the ambient temperature. The same substance might be used effectively at both subzero and normal temperatures because the distinction isn't great [38]. The illustration demonstrates that, regardless of the material's temperature, the compression rating of the CF/epoxy hybrid specimens is lower than their tensile strength. The disintegration process produces a significant quantity of fresh, uneven surface space, which is necessary for composite substances to have compressive properties. The CF/epoxy hybrid composite's compression examination demonstrates that damage mechanisms under compression stress include kink zone development, micro-buckling, and rotation of polymer strands [40]. Because the interface bond's strength is stronger at lower temperatures, the compressive strength rises. Considering the strength under compression at ambient temperature and a subzero temperature of −60 °C, the empty CF epoxy specimens exhibit a substantial rise in compression ability, while the specimen containing 3 weight percent of $4ZnOB_2O3H_2O$ records the smallest increase (11.20%). At a subzero freezing point of −60 °C contrasted with ambient temperature, the compressive value of samples containing 3 wt % SiO_2 rose by 15.85%.

3.2.4 ILSS Behaviour

A mechanical parameter known as interlaminar shear strength (ILSS) assesses a composite material's ability to withstand shear stresses that are placed among its various layers, or laminae. Interlaminar shear strength describes a material's capacity to withstand the stresses that seek to move a single layer of a natural fibre or nanofiller-based composite across another at the contact. ILSS is examined in several samples of empty, SiO_2-filled, and $4ZnOB_2O3H_2O$ samples. At a crosshead rate of 2 mm/min, the ILSS for the hybrid material was measured at both ambient temperature

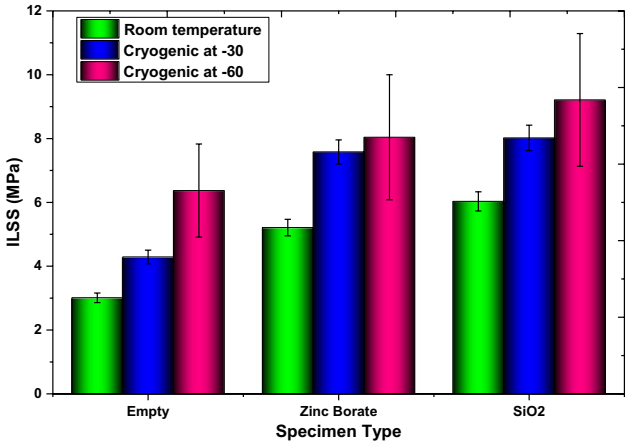

Fig. 7 ILSS behaviour of CF/nano filler-based hybrid composites under different temperature conditions

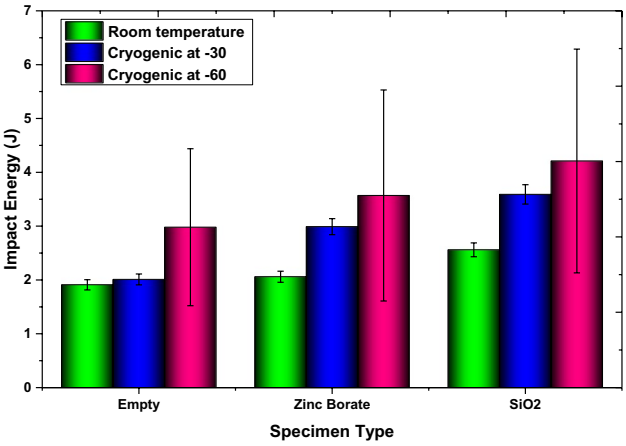

Fig. 8 Impact strength of CF/nano filler-based hybrid composites under different temperature conditions

and below zero (−30 °C and −60 °C). We can observe from the figures that follow that the ILSS rises in temperature below zero. The matrix's phase stiffening that occurs at low load rates like 2 mm/min increases its shear strength. Following prolonged exposure to liquid nitrogen, the blended samples' capacity for asymmetrical expansion and debonding rises [41]. The findings further demonstrate that the fluctuation of the ILSS for an empty CF/epoxy sample through ambient temperature to below zero (−60 °C) is only 12%. The experiment's findings reveal that regardless of the fillers, there is no discernible distinction between the ILSS values tested at ambient temperature and at freezing temperatures, as illustrated in Fig. 7.

3.2.5 Impact Strength

The use of organic fibre-strengthened composites as structural components is restricted by their poor impact resistance. In this section, Charpy impact testing is done to see how the CF/epoxy hybridization laminate responds to impacts at both subzero and ambient temperatures. The 3 wt% SiO_2-filled specimens exhibit the greatest increase in energy absorption (28%) at below-freezing temperatures of −60 °C, whereas the empty CF/epoxy laminated materials exhibit a small rise in absorbing radiation at the same temperatures. Due to their bigger area of surface per volume fraction and superior adherence, the 3 wt% SiO_2-loaded samples demonstrate greater durability against deformation and absorb the most force before breaking at the concentrated impact zone [42]. The findings of energy absorption for different loaded and empty hybrid layers are shown in Fig. 8. In comparison to the sample evaluated at ambient temperature, the filled CF/epoxy hybrids evaluated at below-freezing temperatures demonstrated higher impact resistance. At temperatures below freezing, the dynamic properties of the free volume and voids of the nanoparticle-filled and hardened epoxy remain unaltered, providing sufficient room for molecules to deform themselves and absorb a greater proportion of the energy after impacts. Additionally, hydroxyl groups in the matrix's structure improve the bonding of hydrogen, increasing the interactions between molecules and allowing for greater levels of absorption of energy at lower temperatures compared to ambient temperatures. The detrimental mechanisms for failures in impacts are fibre breaking and matrix fracturing [43].

A few aspects may be taken into consideration in order to analyse the observed pattern of bigger error bars in ILSS and impact strength at −60 °C under cryogenic settings relative to tensile, flexural, and compression properties:

The mechanical characteristics of certain materials, such as hybrid composites, can be greatly affected by cryogenic temperatures. This is especially true for ILSS (interlaminar shear strength) and impact characteristics. Low temperatures cause a variety of behavioural variations in substances, including modifications to their mechanical reactions. This phenomenon holds particular significance in the context of hybrid composites, wherein distinct constituents, such as $4ZnO{\cdot}B_2O3{\cdot}H_2O$ and Nano SiO_2, can display diverse behaviours when subjected to cryogenic temperatures. The way reinforcing fibres and the matrix interact in composite materials is a key factor in defining their mechanical characteristics. Temperature fluctuations may cause alterations in interlaminar bonding at the matrix-fibre interface, which might therefore have an impact on ILSS. Certain materials may undergo a change in behaviour from ductile to brittle at cryogenic temperatures, which can affect the composite's fracture toughness and capacity to withstand crack propagation [44].

Low-temperature thermal contraction can cause internal tensions to arise in the hybrid composite because various

components may contract at different rates. Changes in microstructure and mechanical response may result from this shrinkage in conjunction with phase shifts in the components of the composite. Additional effects of cryogenic environments include microcracking and delamination of the inside of the composite structure, which result in weak areas in the material and can drastically lower impact strength and ILSS. In general, the statistics for impact strength and ILSS are greater than the data for flexural, tensile, and compression characteristics. Greater fluctuation from higher values can lead to bigger error bars. At very low temperatures, composites may behave differently, with impact strength and ILSS being affected differently than tensile, flexural, and compression characteristics. The higher error bars at -60 degrees would suggest that it is more difficult to forecast how well composites will function in these kinds of circumstances [45].

3.3 Flammability Testing

The images of the experimental setup showing both the vertical and horizontal flammability tests are shown in Fig. 9. Table 2 shows the outcomes of the manufactured samples' both vertical and horizontal combustibility tests. Equation 1 was used to get the combustion rate. While the specimens burned in the horizontal test much more slowly than they did in the vertical flame examination owing to the slower combustion circulation, it can be seen that the outcomes of both tests showed a similar pattern. In addition, SiO_2 and $4ZnOB_2O3H_2O$ are followed by a reduction in combustion rates. The burning rate was reduced as a result of the use of nanofillers [33]. This is due to zinc borate's promising flame-retardant qualities, which prevent flame spread and aid in flame extinguishment. Whenever subjected to elevated temperatures, zinc borate releases water vapour; this is one of the main ways it functions as a flame retardant. The substance is cooled by this water vapour, which can help reduce the material's boiling point and delay burning. Inflammable gases may be diluted by the water vapour that has been produced, lowering their potential for ignition and flame propagation. This will aid in lowering the rate at which the materials burn. Zinc borate exhibits endothermic responses if heated, which is another major factor. This implies that in order to breakdown, it needs heat from its surroundings, particularly the flames. This heat absorption assists in cooling the substance and the surrounding region, which slows the spread of the fire [46].

Zinc borate is a typical addition utilised in a variety of substances to increase their combustibility protection since it is both a retardant and a smoke suppressor. It can cause the outermost layer of the material to form a barrier and emit water vapour, thereby helping to avoid or postpone burning. A fire's ability to grow, produce smoke, and generate heat may all be effectively controlled by zinc borate. When resistance to fire is a problem, it is frequently used in plastic bottles, rubber, clothing, timber, and various other materials.

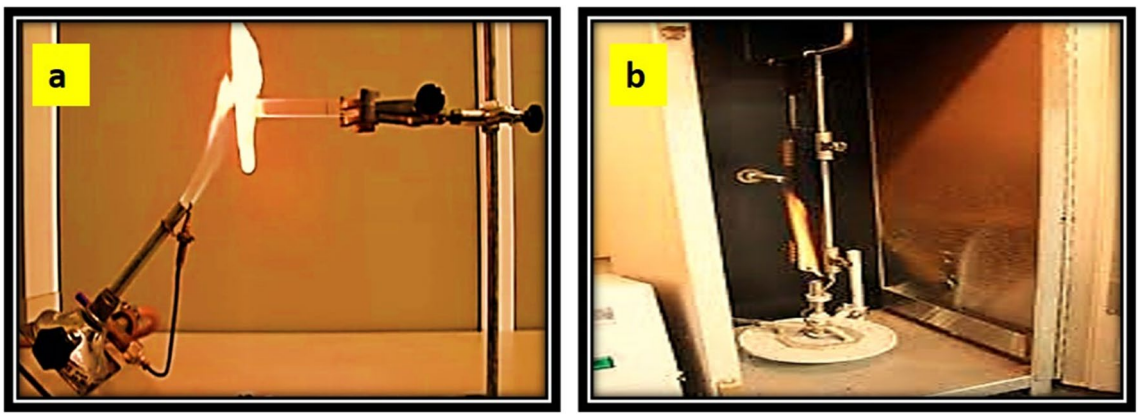

Fig. 9 Photographic images of (**a**) Horizontal burning; (**b**) vertical burning testing of CF/nanofiller based hybrid composites

Table 2 Flammable results of CF/nano filler-based hybrid composites

Sample	Flame travel time (s)		Flame travel distance (mm)		Burning rate (mm/min)		Difference in burning rate (mm/min)
	H	V	H	V	H	V	V-H
CF/Epoxy	170	49	75	75	26.47	91.84	65.37
CF/Epoxy/Zinc borate	630	210	75	75	7.14	21.43	14.29
CF/Epoxy/SiO_2	460	163	75	75	9.78	27.61	17.82

According to the data, the zinc borate's fire-retardant qualities are more closely related. By producing water vapour and creating a barrier of protection, it can actively engage in the process of burning and limit the development of flame [47]. It is frequently used in items that need strong flame-retardant activity. In the same way that zinc borate is a flame retardant but silicon dioxide (also known as silica) is not. It does not actively take part in the burning process or put out flames. Rather, it is used to enhance the substance's thermodynamic and mechanical characteristics, increasing their heat and fire resistance. By lowering thermal conductivity and enhancing rigidity at elevated temperatures, silica can improve a substance's total resistance to fire. In conclusion, zinc borate would seem to be a preferable option if you're seeking a substance with effective flame-retardant qualities. Yet, silicon (SiO_2) could be a better choice if your goal is to increase a material's overall protection against fire by strengthening its thermal and mechanical characteristics [20]. Figure 10 shows the burning rate of composites with both horizontal and vertical positions.

3.4 Water Absorption and Swelling Behaviours

Figure 11 shows the amount of moisture as a function of soaking duration as well as the percentage weight growth of the specimens over time. The interface adhesion of the fibre reinforcement, additives, and matrices, as well as any vacancies in the composite materials, all play a role in how well the materials absorb water. Typically, the very first phase of absorbed water involves a sharp rise. Up until a saturation point, the amount of water absorption rises. When contrasted with filled composite materials, water uptake in CF/epoxy hybrids is greater in the absence of filler. The microchannels that increase the uptake of water by causing dispersion in filled polymers have been

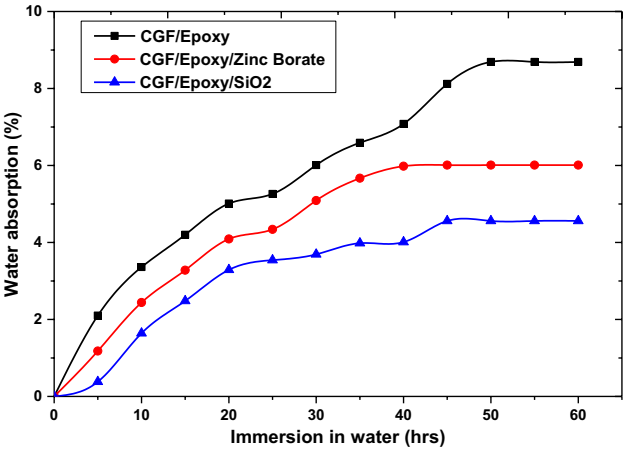

Fig. 11 Water absorption behaviour of CF/Different nanofiller based hybrid composites

filled to a smaller degree. Therefore, as stated, composites containing SiO_2 and $4ZnOB_2O3H_2O$ show only negligible water absorption. As the mesoscopic volume that is free fluctuates with accumulation, the filler's being evenly dispersed within the matrix of polymers has an impact on how much water is absorbed. Compared to hybrid materials without any nanofiller, those strengthened with nanofiller saw considerably reduced weight gains after soaking. Silicon dioxide gained weight at a rate of 4.56% compared to $4ZnOB_2O3H_2O$'s 6.01%. This suggests that the SiO_2 nano-fillers function as an efficient water barrier, lowering the likelihood of liquid penetration. Water molecules permeate into the material through a convoluted channel that is made possible by the large aspect ratio of the nano-fillers. Such nano-fillers restrict the neighbouring epoxy's intermolecular movement, which slows down the

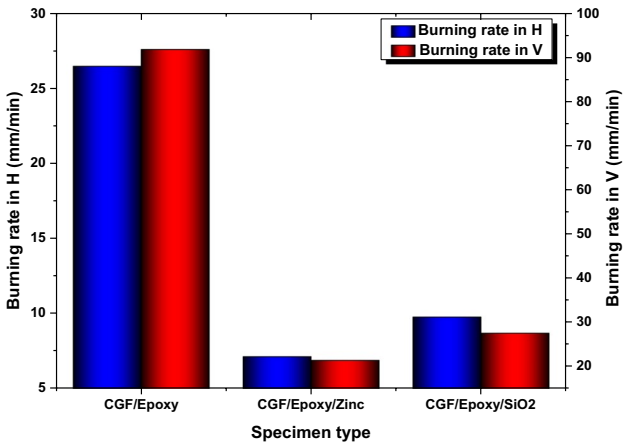

Fig. 10 Shows the burning rate of composites with both horizontal and vertical positions

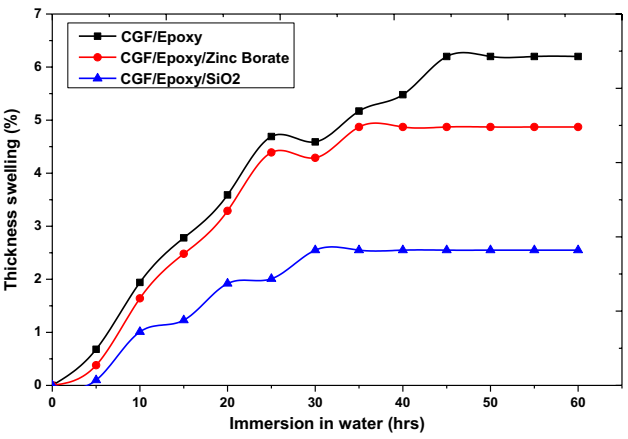

Fig. 12 Thickness swelling behaviour of CF/Different nanofiller based hybrid composites

polymeric strands' relaxation and prevents the passage of microscopic water molecules into the nanocomposite [48].

The impact of the diffusion of water on the expansion of the thicknesses of CF and hybrid laminated materials was examined using a thickness swelling test (Fig. 12). The findings of the swelling experiment corresponded with the outcomes of a water uptake experiment; the swelling was bigger as more water was absorbed. Less swelling was seen with the inclusion of nano-fillers; although SiO_2 NP showed swelling of 2.55%, zinc borate showed swelling of 5.20%. This is due to the fact that silicon dioxide, in its most prevalent forms like quartz, is not greatly permeable and possesses a poor capacity to absorb moisture. Due to its porous nature, amorphous silicon dioxide, which is frequently employed as a filler or reinforcement in many substances, can slowly absorb some water. Although, in comparison with other substances, silica typically has a modest absorption rate [49].

3.5 Microstructural Analysis

Through the use of a scanning electron microscope (SEM), it is possible to examine the adherence of CF to an epoxy matrix, which serves as a gauge of the damage to the hybrid. The three-point bend testing sample's destruction process is shown in the SEM photos (Fig. 13). The sample was tested both at ambient temperature and after being exposed to a nitrogen solution. Figure 13a demonstrates how matrices with nanoparticles on the fibre improve the bonding between the fibre and matrices. Figure 13b depicts the matrix fracturing of the CF/epoxy hybrid specimens during exposure to liquid nitrogen. Figure 13c and d depict fibre pullout and fibre/matrix deformation during ambient temperature bending evaluation, respectively [41].

Due to the different expansion and contraction rates of the fibres and matrices, disintegration and microcracking occur at higher rates and at lower ones, respectively. It is clear that the shrinkage discrepancy at cooler temperatures is produced by the heterogeneity of both the fibre and substrate. Figure 13d inclusion of epoxy and filaments shows that both the matrix and fibres of the hybrid laminate have a strong interface connection. Higher mechanical durability is a consequence of the stronger fibre-matrix interaction. The interface rigidity and adhesive capacity are improved by the filler reinforcements. The stiff filler reinforcements improve the flexural strength of the hybrid material [50]. In samples exposed to freezing temperatures, substrate fractures reveal a stiffening and fragile structure that contributes to the CF/epoxy hybrid composite's increased strength as a material.

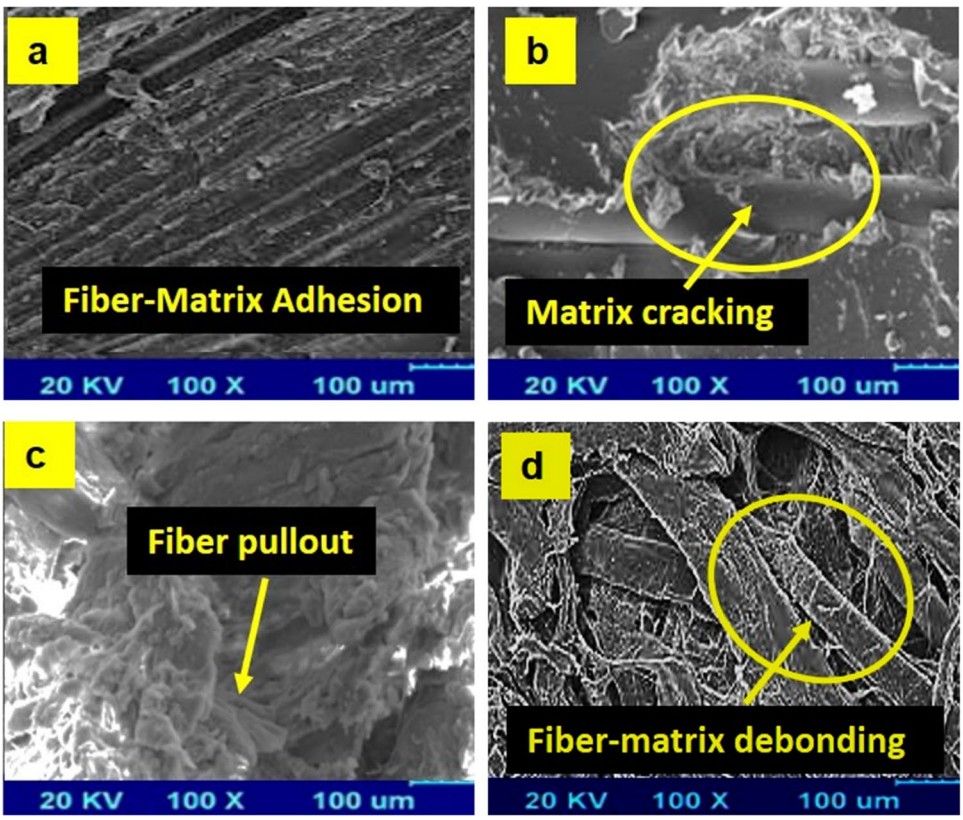

Fig. 13 Microstructural images of CF/ SiO_2 based hybrid composites under (**a**) Room temperature; (**b**) -30º C of cryogenic conditions; (**c**) and (**d**) −60 °C of cryogenic conditions

4 Conclusion

In conclusion, the thorough investigation into the mechanical and flammability properties of hybrid materials based on Crown flower/Nano SiO_2/$4ZnOB_2O_3H_2O$ in cryogenic situations has provided invaluable insights into their prospective uses in hostile environments. We have discovered via a series of thorough experimental examinations that exposure to cryogenic temperatures significantly improves the mechanical characteristics of the hybrid composites, including tensile strength, flexural strength, and impact resistance.

- The presence of nanoSiO_2 and $4ZnOB_2O_3H_2O$ was confirmed in CF/nanofiller-based hybrid composites through XRD and FTIR analysis. As a consequence, the findings demonstrated that the product has a monoclinic crystal state ($4ZnOB_2O_3H_2O$).
- Nano SiO_2 and $4ZnOB_2O_3H_2O$ additions have shown to be efficient in strengthening the composite matrix, which has helped to enhance its mechanical properties. The findings suggest that because of the small size and huge surface area of nanofillers, there may be a variety of benefits to their introduction. Tensile strength improves by 15.87% for the unfilled sample at −30 °C, 9.23% for the specimens filled with 3 weight percent $4ZnOB_2O_3H_2O$, and 13.65% for the specimens loaded with 3 weight percent SiO_2.
- It can strengthen the link between the structure of the material and the filaments, thereby decreasing the likelihood of cracks spreading and increasing the material's resilience to distortion. Additionally, by serving as stress-transferring connections, they help distribute the stress more uniformly throughout the substance.
- Furthermore, such hybrid composites' flame-retardant qualities have shown an impressive capacity to endure flammability issues even at cryogenic temperatures, indicating their feasibility for employment in settings where fire dangers are a concern.
- In comparison to SiO_2, zinc borate exhibits a low burning rate, i.e., 7.14 mm/min. Whenever subjected to elevated temperatures, zinc borate releases water vapour; this is one of the main ways it functions as a flame retardant. The substance is cooled by this water vapour, which can help reduce the material's boiling point and delay burning. Inflammable gases may be diluted by the water vapour that has been produced, lowering their potential for ignition and flame propagation.
- Silicon dioxide gained weight at a rate of 4.56% compared to $4ZnOB_2O_3H_2O$'s 6.01%. This suggests that the SiO_2 nano-fillers function as an efficient water barrier, lowering the likelihood of liquid penetration. Less swelling was seen with the inclusion of nano-fillers; although SiO_2 NP showed swelling of 2.55%, zinc borate showed swelling of 5.20%.

Nevertheless, it is crucial to recognise that further study is needed. Validating the long-term survivability and performance of these hybrid composites requires extensive long-term stability investigations, in-depth microstructural analyses, and more thorough testing under a wider variety of cryogenic temperatures. As these materials are put into practise, environmental impact studies should also be taken into account. In conclusion, this work highlights the promising potential of hybrid composites based on Crown flower/Nano SiO_2/$4ZnOB_2O_3H_2O$ for improving mechanical and flammability properties in cold conditions. The findings of this study pave the way for future developments in material research by providing answers to problems caused by harsh circumstances and aiding in the creation of safer and more resilient materials for use in a variety of sectors.

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Ramesh Velumayil and Ranjith Kumar R: Investigation.
Elil Raja D, Nagaraj M and John Presin Kumar A: Testing and Evaluation.

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Declarations

Yes.

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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