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Fire Behavior of bio-sourced composites under Varied Heat Flux Levels

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Abstract **-** This study aims to enhance the understanding of the fire behavior of bio-sourced composite materials for potential applications in aeronautics and automobiles. Experimental tests were conducted using the NexGen burner, designed according to FAA requirements for fire certification, and a cone calorimeter to assess medium-scale fire behavior. The research also focused on developing flax and banana-reinforced Greenpoxy composite materials, addressing concerns related to the non-biodegradable nature of synthetic composites and global warming. The green biocomposites manufactured through the Vacuum Assisted Resin Transfer Molding (VARTM) method, underwent thermal-physical characterization. Thermal degradation studies involved three heat flow densities (20, 35, and 50 kW/m²), analyzing fire performance indices, mass loss, surface temperatures, and gaseous emissions. The results revealed that biocomposites exhibited distinct thermo-physical behavior on medium and large scales, with flax-based materials showing superior thermal properties on a medium scale and banana-based materials demonstrating the opposite but interesting trend on a large scale. The study highlighted the size-dependent nature of bio-sourced material thermal properties. This research contributes valuable insights into the fire performance of bio-sourced composites, addressing critical aspects of materials.

Keywords**:** Green biocomposites, VARTM, NexGen Burner, Cone calorimeter, Thermal properties, Fire safety.

1. Introduction

The development and utilization of composite materials, particularly in the transport sector, have been on a significant rise due to their exceptional mechanical properties, lightweight nature, and high performance in diverse environmental conditions. Among these composites, biosourced and synthetic types have gained considerable attention due to their potential for sustainability and high-performance characteristics [1]. However, their application brings forth critical challenges, especially concerning their fire behavior. Understanding the fire behavior of these composites when subjected to various heat flux densities is crucial for ensuring safety and compliance with fire standards in the transport industry. The transport industry, encompassing automotive, aerospace, maritime, and rail sectors, constantly seeks materials that offer not only structural efficiency and weight reduction but also economic viability and environmental sustainability. Biosourced composites, made from natural fibres and bio-based resins, with a polymer matrix, have emerged as leading materials meeting these demands. Their adoption promises enhanced fuel efficiency, reduced carbon emissions, and a lower environmental footprint, aligning with global sustainability goals. However, the integration of these materials into the transport sector's infrastructure and vehicles presents a complex challenge: ensuring robust fire safety standards without compromising their beneficial properties [2].

To address these challenges, experimental studies focusing on the fire behavior of biosourced composites under varied heat flux densities are essential. Such studies provide invaluable insights into the ignition, combustion, and flame spread characteristics of these materials. By examining composites on a medium (cone calorimeter) and large scale (NexGen burner), researchers can better understand the real-world implications of fire incidents in the transport sector. These experimental investigations help in the development of models that predict fire behavior, guiding the design and manufacturing of safer composite materials.

The impact of different heat fluxes on the fire behavior of composites is a critical area of study. Heat flux, a measure of the rate of heat energy transfer per unit area, significantly influences the ignition time, flame spread rate, and overall combustion dynamics of composites. By subjecting biosourced to various heat flux densities, researchers can simulate a range of fire scenarios, from slow smoldering to rapid flashover conditions. This information is pivotal in designing composites that are not only high-performing and sustainable but also exhibit enhanced fire retardancy [3].

In this study, two biodegradable materials based on bio resin reinforced with flax and banana were manufactured using the Vacuum Assisted Resin Transfer Method (VART Method). In order to provide elements of understanding, two experimental devices aimed at studying the thermal behavior of a composite material were used: (a) a cone calorimeter assimilated to a intermediary scale and (b) the NexGen burner assimilated to a real-life fire condition. The overall objective is to study the effect of three heat flux densities (20, 35 and 50 $kW.m^{-2}$) and intumescent flame retardant on the thermal, physical and chemical properties of the flax and banana laminate composite materials manufactured.

2. Materials, Equipment and Methods

Flax fibres were selected as natural fibre reinforcement for the production of the composite material. The flax fibres used had a 2x2-twill weave structure with an area density of 550 $g.m^{-2}$ and a linear density of 0.0045 $g.m^{-1}$. The woven fabric was purchased from Eco-technilin, France.

The banana was chosen as the natural fibre reinforcement for the production of the composite material and was inspired by its special properties and possible applications as revealed in Figure 1 (right). The banana fibres used in this study exhibited remarkable properties, including an areal density of 420 $g.m^{-2}$ and an exceptionally low linear density of 0.00004 $g.m^{-1}$.

A bio-based epoxy resin called Greenpoxy 56, which contains a bio-based content of 56%, was selected as the matrix for binding the natural fibre reinforcement. It played a crucial role in transferring loads between fibres and giving the composite its final shape. The specific bio-based epoxy resin used, Infugreen 810, was obtained from Sicomin, France. To initiate the curing process of the epoxy system, SD 8822 was used as the hardener/hardener. The epoxy was infused to facilitate the molding of the fibre-reinforced composites (FRC) laminates.

A fire-resistant coating used consists of ammonium polyphosphate-tris-2-hydroxyethyl isocyanurate (APP-THEIC) and boric acid (BA) which releases water when heated, acting as fuel diluters, heat absorbers, and blowing agents for the intumescent coating. Ammonium polyphosphate reacts with boric acid, producing borophosphate, enhancing char stability. The combination of APP and boric acid significantly delayed the steel substrate's temperature rise to 400°C, ensuring a well-adhered char layer.

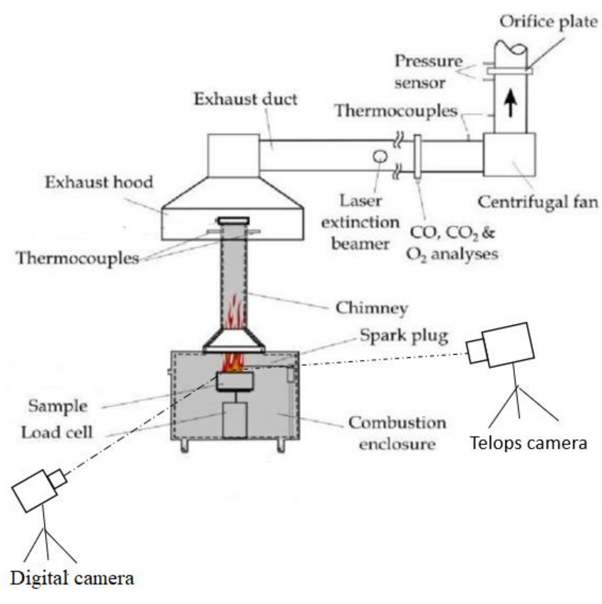

Fig. 1: Experimental schematic set up for cone calorimeter analysis

2.1. Biocomposites preparation

The production of green biocomposites based on flax and banana reinforcements using Greenpoxy 56 and an amine hardener was carried out through the Vacuum Assisted Resin Transfer Molding (VARTM) method at INSA CVL Bourges. After the fabrication process, the green biocomposites were carefully trimmed and cut to meet the desired specifications and final dimensions i.e. 100 mm x 100 mm for the cone calorimeter test and 500 mm x 500 mm for the Vesta NextGen burner. The final thickness of the flax and banana green biocomposite obtained are 5.5 ± 0.1 mm and 5.45 ± 0.1 mm respectively. A fire-resistant film coating was then applied to the surface of the green biocomposite.

2.2. Cone calorimeter

The experimental setup closely followed the configuration guidelines outlined in the ISO 5660-1:2015 standard as shown in Figure 2. The cone calorimeter used was manufactured by Deatak in the USA.

Additionally, the Telops infrared camera was strategically positioned to measure the thermal degradation camera. To ensure repeatability, three samples were examined per test [4].

2.3. NexGen burner

This section introduces the NexGen burner, part of the Fire VESTA experimental setup, designed for assessing material degradation. The test bench, tailored for certification tests resembling aircraft engine incidents, evaluates material thermal degradation exposed to a kerosene/air flame with specific temperature and flow parameters. Components critical to the burner's functionality, including air and fuel supply systems, are outlined. The sample, held securely in a holder with a deflector, undergoes pinching to ensure stationary testing conditions. The deflector prevents flame bypass, protecting the sample's rear face. A hood, located on the upper part of the test bench, extracts combustion and pyrolysis products. Airflow regulation, managed by a pressure regulator and sonic choke, induces swirling motion with a stator and turbulator for enhanced turbulence. Combustion initiation employs a spark plug activated by a 10-kV voltage at the start of testing. Additionally, the heat flux of the flame was calibrated using the Captec sensor, a Telops camera was used for the surface temperature determination of the biocomposites, a gas analyzer for the gas emission measurement, and a digital camera to monitor the entire process (the setup is shown in Figure 2) [5].

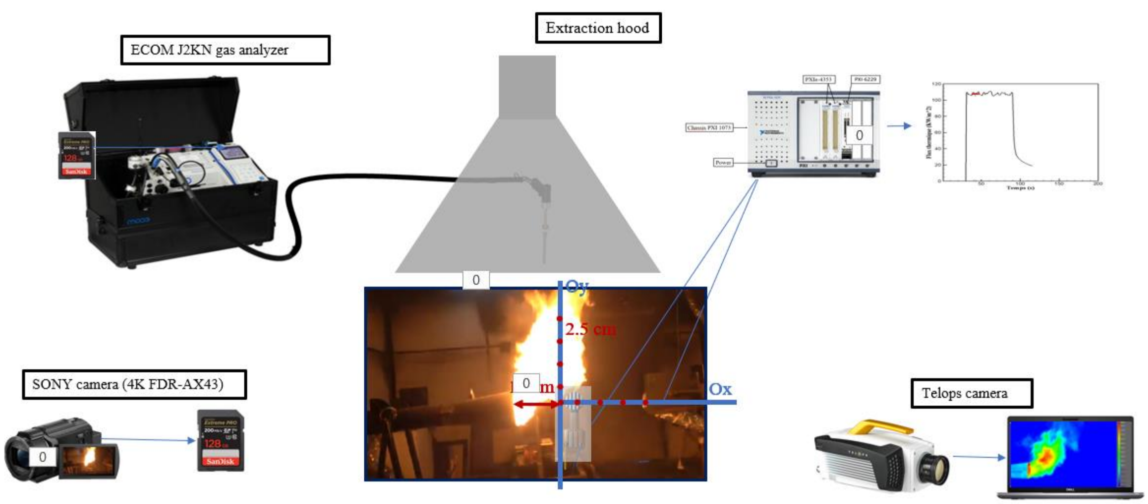

Fig. 2: The setup of NexGen burner during test

2.4. Methodology

Cone calorimeter: The prescribed standard test of the cone calorimeter allows for authentic degradation scenarios by covering a range of heat fluxes, specifically at 20, 35, and 50 $kW.m^{-2}$. The cone calorimeter provides several significant measurements, including heat release rate (HRR), mass loss rate (MLR), Time to Ignition (TTI), Carbon dioxide (CO_2) concentration, Carbon monoxide (CO) concentration, and Smoke production rate (SPR). The test sample's HRR is computed with Equation 1. HRR relies on the assumed heat produced per unit mass of oxygen (13.1 MJ/kg O_2), the oxygen depletion factor, and the mass flow rate in the exhaust duct (from Equation 2). The total smoke production (TSP) in square meters is obtained by integrating the SPR in square meters per second, as indicated by Equation 3.

$$\dot{Q} = 1.10\,(E)(m_e)(X_{O_2}^{i})\left[\frac{\emptyset}{1.105\emptyset + (1-\emptyset)}\right] \quad (1)$$

$$\dot{m}_e = C\sqrt{\frac{\Delta P}{T_e}} \quad (2)$$

$$TSP = \int_0^t SPR\, dt \quad (3)$$

Where
$\dot{Q}$: Heat release rate
E: Activation energy
m_e: Mass of the material
$X_{O_2}^{i}$: Initial oxygen concentration
$\emptyset$: Mass fraction of the combustible material
$\frac{\emptyset}{1.105\emptyset + (1-\emptyset)}$: Correction factor
m_e: mass loss rate, t: time
$C\sqrt{\frac{\Delta P}{T_e}}$: Total heat of combustion
TSP: Total smoke production
SPR: Smoke production rate

NexGen *burner:* The Captec device with a measuring range of 200 $kW.m^{-2}$ and a sensitivity of 50 $mV/(kW.m^{-2})$ was used to calibrate the heat flux of the kerosene/air flame at 20, 35, and 50 $kW.m^{-2}$. A gas analyzer, ECOM-J2KN Pro, connected to the NexGen setup collects gas emission species. The experiment was based on the optimum fuel flow rate of 0.0019 $kg.s^{-1}$, air flow rate of 0.02845$kg.s^{-1}$, and equivalence ratio of 1.03 to assess the combustion performance of the flax and banana green biocomposites. These samples were placed 73, 62, and 43 cm away from the cone burner to obtain the heat flux conditions of 20, 35, and 50 kW/m² respectively.

Thereafter, the biocomposite combustion test was performed until complete degradation was observed. The mass loss of the composite sample is measured using a SCAIME AG3 center support load cell located under the sample holder with an uncertainty of ±5g.

The Ecom-J2KN Pro analyzer operates with the non-dispersed infrared principle (NDIR) with the accuracy of ±0.2%, ±2%, and ±5% for O_2, CO, and CO_2 respectively. The surface temperature was also measured with the help of the Telops IR camera which operates via the use of a filter to ghost the flame and record the front-face temperature of the biocomposite samples.

3. Results and discussion

Cone calorimeter results:

Table 1 summarizes cone calorimetry results for green epoxy composites reinforced with flax and banana fibers. Tests were conducted at heat flux levels of 20, 35, and 50 $kW.m^{-2}$, offering a comparative overview of composite performance under different thermal stress conditions.

Table 1: Cone Calorimeter Evaluation of Flax and Banana Green Biocomposites under Varying Heat Flux

	Flax_G-poxy			**Banana_ G-poxy**		
Parameters	**20** $kW.m^{-2}$	**35** $kW.m^{-2}$	**50** $kW.m^{-2}$	**20** $kW.m^{-2}$	**35** $kW.m^{-2}$	**50** $kW.m^{-2}$

Tig (±2)/s	144.9	67.0	36.0	125.0	55.7	32.5
pHRR (±5)/kW.m^{-2}	495.4	693.7	823	753.1	761.9	908.6
pHRR$_t$ (±0.5)/s	319	290	177	154	263	145
THE (±3)/MJ	87.5	102.8	114.1	83.8	122.2	129
ARHE (±4)/ kW.m^{-2}	393.1	611.9	771.7	513	790.4	1066.6
FIGRA (±0.2)/kW.m^{2}s^{-1}	1.56	2.39	4.65	2.9	4.89	6.27
FPI (±0.1)/kW.m^{2}s^{-1}	3.44	10.35	22.86	6.02	13.68	27.96
Av-CO_2Y (±0.002)/kg.kg^{-1}	3.08	4.004	5.638	3.00	4.294	6.133
Av-COY (±0.005)/kg.kg^{-1}	0.086	0.1142	0.132	0.18	0.20	0.211
Residue (±0.5)/g	6.17	5.19	0.52	6.45	1.49	0.30

Flax_G-poxy = Flax-reinforced Greenpoxy composite
Banana_G-poxy = Banana-reinforced Greenpoxy composite

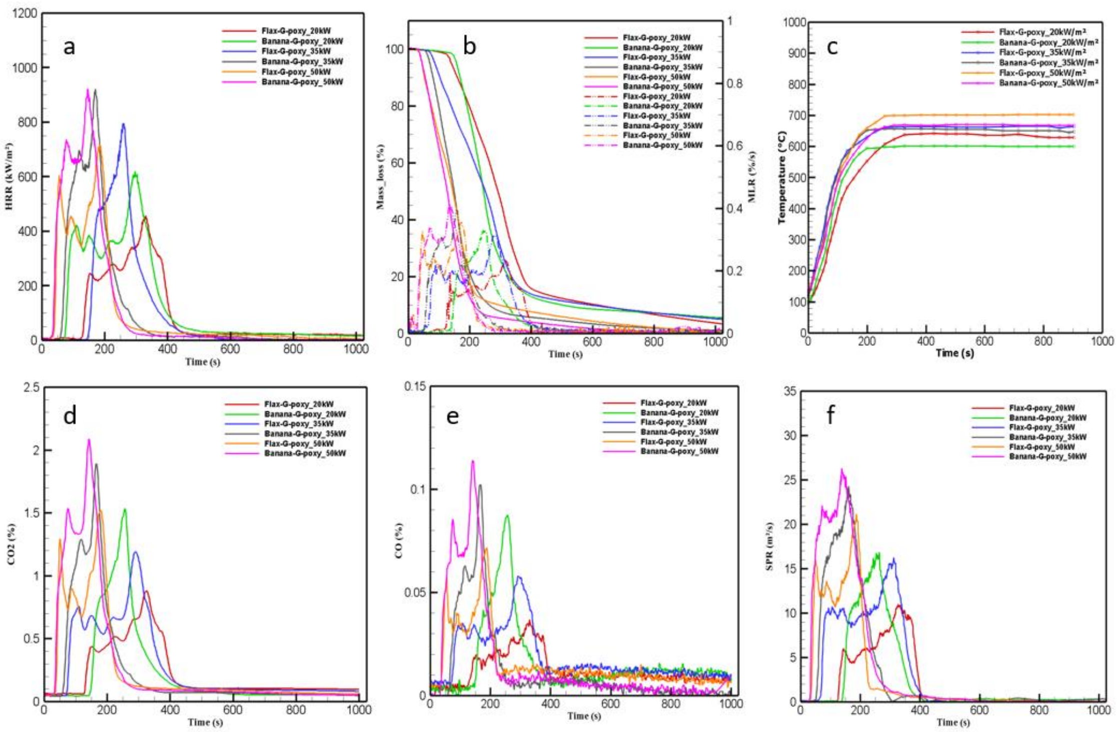

Fig. 3: The fire behavior of the flax and banana biocomposites using the cone calorimeter

The provided graphs display a series of fire behavior tests for Flax-Greenpoxy and Banana-Greenpoxy composites at different heat fluxes (20 kW/m², 35 kW/m², and 50 kW/m²). The discussion is as follows:

a. Heat Release Rate (HRR):

This graph shows the HRR of both composites over time. The peaks indicate the point at which the maximum rate of heat is being released during combustion. Flax-Greenpoxy has a higher peak HRR than Banana-Greenpoxy at all heat flux levels, suggesting flax burns more intensely under these conditions.

b. Mass Loss:

The mass loss graph illustrates the percentage of mass remaining as the composites are exposed to heat over time. The steep decline in the curves indicates rapid mass loss due to combustion or pyrolysis. The dotted lines likely represent the derivative of mass loss, which corresponds to the mass loss rate.

c. Temperature Rise:

Here, the temperature rise over time for each composite at different heat fluxes is shown. All samples appear to reach a plateau, suggesting a balance between heat generation and loss. The Flax-Greenpoxy composite heats up more rapidly than the Banana-Greenpoxy, indicating a possible higher thermal conductivity or a more intense combustion process.

d. CO_2 Production:

This graph shows the production of CO_2 as a combustion product. The Flax-Greenpoxy composite produces more CO_2, especially at higher heat fluxes, which aligns with its higher HRR and indicates more complete combustion.

e. CO Production:

CO production is typically higher in incomplete combustion. The spikes in the graph suggest transient phases where combustion efficiency changes. The overall lower levels of CO for Banana-Greenpoxy could indicate a more controlled or slower combustion.

f. Smoke Production (SPR):

Smoke production rates vary for both materials, with the Flax-Greenpoxy composite generating more smoke at peak times, especially at 50 kW/m². This could be due to the greater amount of material burning or decomposing, as indicated by its higher HRR and CO_2 production.

Overall, the graphs suggest that the Flax-Greenpoxy composite tends to burn more intensely and produce more CO_2 and smoke, while the Banana-Greenpoxy composite shows a more moderate response to the applied heat fluxes. These behaviors are attributed to the chemical composition of the composites and have implications for the use of these materials in applications where fire resistance is a critical property.

NexGen burner results:

The provided graphs depict various fire testing parameters for Flax-Greenpoxy and Banana-Greenpoxy composites at different heat fluxes (20, 35 kW/m² and 50 kW/m²). Here's a concise interpretation of each graph:

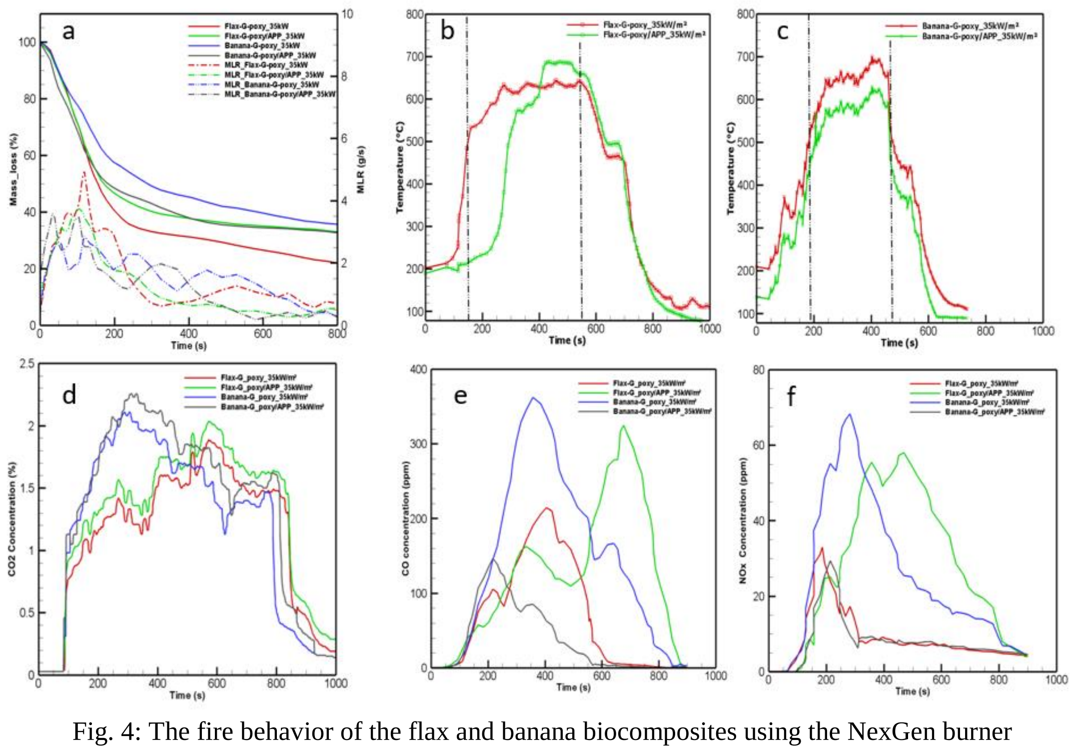

Fig. 4: The fire behavior of the flax and banana biocomposites using the NexGen burner

a. Mass Loss and Mass Loss Rate (MLR):

Both composites show a decrease in mass over time with a more rapid mass loss at higher heat flux (50 kW/m²). The mass loss rate peaks early, indicating the most intense combustion phase occurs at the beginning of the exposure.

b. Temperature Profile (Flax-Greenpoxy, 35 kW/m²):

The temperature sharply rises to a peak and then falls, indicating a quick ignition and burnout phase.

c. Temperature Profile (Banana-Greenpoxy, 35 kW/m² & 50 kW/m²):

A similar trend to Flax-Greenpoxy, but with the peak temperature reached more quickly at the higher heat flux.

d. CO_2 Production:

CO_2 concentrations peak during the main combustion phase, with higher concentrations at 50 kW/m², indicative of more complete combustion at higher heat fluxes.

e. CO Production:

Peaks in CO concentration suggest phases of incomplete combustion. The CO peaks are more pronounced at 50 kW/m², particularly for Flax-Greenpoxy.

f. NOx Production:

NOx concentrations peak and then diminish, with Flax-Greenpoxy showing higher peaks at both heat flux levels, which may be due to higher combustion temperatures or nitrogen content in the material.

Overall, the results suggest that both composites combust more intensely at higher heat fluxes, with more rapid temperature rise, higher peak gas emissions, and faster mass loss. Flax-Greenpoxy tends to show more pronounced reactions in terms of peak temperatures and gas emissions, suggesting a potentially more vigorous combustion process compared to Banana-Greenpoxy.

4. Conclusion

In conclusion, this study addresses significant gaps in the literature on biobased composite materials, particularly in terms of fire resistance and delamination behavior at full scale. By investigating the nuanced responses of natural fiber-reinforced biocomposites to fire conditions at various scales, this research provides valuable insights for accurately predicting their thermo-structural characteristics. The cone calorimeter provides a consistent environment and is generally preferred for fundamental research into material flammability of composite materials. The NexGen burner is used to evaluate the combustion properties of the composite materials under direct flame impingement. It is designed to simulate more realistic fire scenarios, such as those encountered in aircraft engines.

The findings of the results in this study could serve as a valuable database for FDS simulation for future work.

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