Fire reaction properties of flax/epoxy laminates and their balsa-core sandwich composites with or without fire protection

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ABSTRACT

Fire reaction properties of flax/epoxy laminates and their corresponding balsa-core sandwich composites with/without fire protection were evaluated at an applied heat flux of 50 kW/m^2. A strategy for improving the fire reaction properties of flax/epoxy composite materials without adversely altering their intrinsic mechanical properties was investigated. A thin glass fiber veil impregnated with ammonium polyphosphate was bonded onto the composite surface intended for heat exposure. There was no significant change in the time-to-ignition with the introduction of the fire retardant glass fiber veil. While similar PHRR values were measured in both flax/epoxy laminates and balsa-core sandwich composites, the total heat release was significantly higher in the later. It is noteworthy, the heat release rates (including the peak values) measured for fire-protected composites were significantly lower than those measured for their unprotected counterparts. Through-thickness temperature profiles across the sandwich composite revealed the effectiveness of the fire retardant veil in minimizing thermal damage of underlying substrate composites.

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1.0   INTRODUCTION

The need to achieve environmental sustainability in engineering has spurred the development and creation of new engineering composite materials incorporating bio-derived materials [1-7]. Recently, Gallo [6], Duigou [7] and their co-workers successfully manufactured engineering sandwich composites incorporating bio-fibers, bio-resins and wood-based core sections. Regarding sustainability, bio-fibers are a viable alternative to synthetic glass reinforcements due to their desirable material properties including moderately high specific mechanical properties, CO_2 neutrality as well as biodegradability [1-3]. Similarly, issues with the adverse environmental impact and sustainability of petroleum-derived polymer resins have driven an increase in the uptake of bio-resins within the composites industry. However, despite the reported success in the development of bio-derived composites, their deployment in engineering structures is still very limited. Firstly, the structural properties of bio-based engineering composites are generally inferior to those exhibited by their counterparts incorporating petroleum-based matrices and/or synthetic fiber reinforcements [3]. Consequently, bio-derived composites are limited to non- or semi-structural engineering applications. Second and most importantly, due to their flammable nature [8], composites incorporating bio-derived constituents pose increased fire risks when used in engineering applications threatened by fire.

Engineering sandwich composites have almost always utilised glass or carbon fiber-reinforced polymer face skins together with light-weight core materials such as polymer foam, polymer-based honeycombs, cork or balsa. The intrinsic flammability of the polymer matrices and traditional non-metallic core sections pose increased fire risks. That is, the incorporation of bio-fiber reinforcements which are inherently flammable can only serve to exacerbate the fire risks already limiting the utilization of glass or carbon fiber/polymer composites in fire-prone environments. The subjects of fire reaction and resistance of composites incorporating highly flammable bio-derived constituents are worth investigating. Before these intrinsically flammable composites can be deployed in thermally demanding environments, strategic solutions that will provide adequate fire retardation and increased fire resistance need to be developed. Recently, Kandola and

Kandare [9] reviewed wide-ranging fire retardation strategies shown to significantly improve both the fire reaction and fire resistance of synthetic fiber/polymer composites. A considerable number of fire protection strategies reported in this literature review may be adapted to improve the fire performance and fire structural survivability of composites incorporating highly-flammable plant-derived constituents.

A common strategy to improve fire reaction properties of fiber/polymer composites involves the modification of the matrix via the inclusion of micro-sized fire retardant additives. Several authors successfully demonstrated improvements in fire reaction properties of polymer composites incorporating fire retardant additives [9-17]. However, in most cases, fire safety ratings are achieved at elevated fire retardant loading concentrations. Inadvertently, high fire retardant loadings lead to the degradation in ambient-temperature mechanical properties of fire-retarded fiber/polymer composites [18]. To circumvent this problem, fire retardation strategies precluding the inclusion of fire retardant additives throughout the fiber/polymer composite matrix are preferred. One of these strategies involves the application of insulative materials on heat-exposed surfaces of composite structures. Recently, the author together with his colleagues evaluated the fire reaction behaviour and fire structural performance of surface-insulated fiber/polymer composites [19-22]. The application of insulative silica-based materials or fire retardant coatings onto the heat-exposed surfaces of fiber/polymer composites was found to retard flaming-combustion. Physically restricting the fire retardant additives to the heat-exposed surface significantly improves the fire reaction properties and fire structural performance of composites without adversely affecting their ambient-temperature mechanical properties.

In this paper, the fire reaction properties of epoxy composites incorporating flax fibers and balsa core were evaluated. The fire retardation efficacy of an insulative mat recently developed by our collaborators within the Cooperative Research Centre for Advanced Composite Structures (CRC-ACS), Australia was investigated. This insulative mat is a glass fiber veil impregnated with a char-forming additive, ammonium

polyphosphate (APP), using proprietary technology. The fire retardant veil was bonded onto the surface of flax/epoxy laminates or balsa-flax/epoxy sandwich composites and is therefore not expected to adversely affect ambient-temperature mechanics of these materials. This study was performed with the objective to improve fire reaction hence fire structural performance of fibre/polymer composites incorporating highly flammable bio-derived constituents. Due to structural property (strength and stiffness) limitations, bio-fibre/polymer composites are restricted to semi-structural applications in the civil engineering. Therefore, the fire reaction properties of flax/epoxy laminate and balsa-flax/epoxy sandwich composites were measured under cone calorimeter heating conditions simulating a well-developed room fire (e.g., an applied heat flux of 50 kW/m^2). In addition to evaluating the fire reaction properties, possible implications of the investigated fire retardation strategy on in-situ and post-fire structural performances of these composite materials are considered. The fire resistance of composites (with/without surface insulation) is discussed in relation to the softening temperature of the composite matrix and the onset temperature of decomposition for the epoxy matrix and/or bio-fibers. The softening temperature is critical in evaluating the survivability of engineering structures during fire while the onset temperature of matrix and/or fiber decomposition is useful in assessing the post-fire structural damage.

2.0   EXPERIMENTAL

2.1   Materials

A room temperature-curing epoxy resin, diglycidyl ether of bisphenol A (DEGBA), a corresponding amine-based hardener and balsa (structural end-grain Baltek SB100; density 150 ± 30 kg/m^3; thickness 25 mm) were all supplied by ATL Composites, Australia. A unidirectional continuous flax fiber fabric (area density = 180 g/m^2) was obtained from Lineo, France. A 500 μm non-woven glass fiber veil incorporating char-forming ammonium polyphosphate as the active ingredient (150 g/m^2) and a pristine glass fiber veil without fire retardant additives were both provided by CRC-ACS, Australia. All the materials were used as received without any purification.

2.2 Preparation of FE Laminate and BFE Sandwich Composites

Flax fiber/epoxy laminate composites (fiber vol. fraction ~30%) were prepared via wet lay-up. The epoxy resin and hardener were mixed in the ratio 4:1 and mechanically-stirred (900 rpm) at room temperature for 15 min. The resultant mixture was de-gassed in a vacuum chamber in order to eliminate air bubbles. The de-gassed resin/hardener formulation was then used to impregnate eight flax fabric layers which were then stacked in a cross-ply configuration to achieve a 4.4 mm-thick laminate. The panel was cured overnight at ambient conditions. Balsa-core flax/epoxy sandwich composites were prepared by uniformly applying the resin/hardener formulation onto balsa surfaces before hand-laying impregnated flax fabrics.

Master panels of flax/epoxy laminate and balsa-flax/epoxy sandwich composites were cut into square plaques (90 mm × 90 mm) for cone calorimeter evaluations. Non-woven glass fiber veils with/without fire retardant additives were then independently bonded onto flax/epoxy laminate or balsa-flax/epoxy sandwich composite surfaces intended for heat exposure using the epoxy/hardener formulation (150 g/m^2). The amount of resin/hardener formulation used in bonding the glass veil onto the surface of the laminate or sandwich composites was calculated such that a 1:1 weight ratio of APP to polymer was achieved within the glass fiber veil (e.g., 50% APP by weight with respect to epoxy). Following the bonding of glass fiber veils onto flax/epoxy laminate or balsa-flax/epoxy sandwich composites, the resultant specimens were left to cure overnight at ambient conditions before post-curing at 100°C for 4 h. Prepared composites are identified as follows: unprotected flax/epoxy laminates (FE); fire-retarded flax/epoxy laminates (FE-APP); unprotected sandwich composites (BFE) and fire-retarded sandwich composites (BFE-APP).

2.3 Fire Reaction Properties of Flax/epoxy Laminate and Balsa-Flax/epoxy Sandwich Composites

The fire reaction properties of flax/epoxy laminates and balsa-flax/epoxy sandwich composites (with/without) fire protection were evaluated using the cone calorimeter in the horizontal testing mode. Test specimens (90 mm × 90 mm × nominal thickness) were subjected to one-sided radiant heating from a cone

calorimeter operating at an applied heat flux of 50 kW/m^2 according to ISO 5660. The heat-exposed surfaces of the test specimens were positioned 25 mm away from the cone heater. Cone calorimeter parameters including the time-to-ignition (TTI), the heat release rate (HRR) and its peak value (PHRR), time-to-PHRR (tPHRR), the total heat release (THR), the mass and the total smoke release (TSR) were measured. During cone exposure, temperature-time profiles were also measured at three locations across the thickness of the sandwich composites. Figure 1 shows the location of the thermocouples: T1, the skin/core interface close to the heat-exposed surface; T2, the neutral axis and T3, the back skin/core interface, respectively.

3.0   RESULTS AND DISCUSSION

3.1   Fire Reaction Properties of Flax/epoxy Laminate Composites

Heat release rate profiles of non-insulated and fire-protected flax/epoxy laminates were measured using the cone calorimeter at an applied heat flux of 50 kW/m^2. The HRR-time data for laminates with (FE-APP) and without (FE) fire protection is shown in Figure 2(a). The heat release rate for FE increases rapidly following ignition to reach an initial PHRR value of 592±40 kW/m^2 after 51 s of exposure as given in Table 1. Following this event, the heat release rate drops rapidly before gradually increasing again to reach a second and higher peak of 616±12 kW/m^2 after 173 s of continuous thermal exposure. As reported by Chai and co-workers [23], this behaviour is not uncommon in bio-derived fiber/polymer composites. A temporary char layer may be formed at the exposed surface of bio-reinforced composites upon heat exposure. In addition to slowing down heat transfer into the underlying composite, the temporary carbonaceous char may also retard mass transportation of combustible volatiles and oxygen into the pyrolysis zone thereby inhibiting flaming-combustion processes. The sudden drop in heat emissions following the first peak event may be attributed to the presence of this temporary thermally-protective char layer. However; with continued thermal exposure, the temporary surface char may degrade thereby exposing the underlying materials. The depletion of the temporary char layer enables the intensification of the flaming-combustion reactions leading to the second heat release peak event.

The fire-retarded laminate (FE-APP) revealed a fire reaction behaviour that is significantly different from that observed for the control laminate (FE). The presence of a char-forming additive within the surface-bound glass fiber veil suppressed flaming-combustion as evident from reduced heat release rates over the first 200 s of thermal exposure. While heat emissions increased rapidly after ignition, the first PHRR event for FE-APP was significantly lower (262 ± 8 kW/m^2) than the value measured for an equivalent feature from the HRR-time profile of FE (592 ± 40 kW/m^2). Following the first PHRR event observed after 62 s, a slight dip in the heat release rate followed by a gradual increase to reach the second peak event after 211 s of continuous thermal exposure was observed. The second HRR peak value in FE-APP (485 ± 47 kW/m^2) is 21% lower than the corresponding value measured for the control laminate (616 ± 12 kW/m^2). Previous research conducted on glass/epoxy laminates of similar thickness under the same cone calorimeter conditions revealed that a thin surface layer of tricresyl phosphate-based coating significantly reduced flaming-combustion intensities [19]. The PHRR measured for the tricresyl phosphate-protected glass/epoxy laminate was 20% lower (584 kW/m^2) than the value measured for the respective control laminate (733 kW/m^2) [19]. It is clear from this current study that the presence of flammable flax reinforcements does not necessarily deplete the fire retardation efficacy of phosphate-based (e.g., char-promoting) formulations.

The dissimilarity in flaming-combustion intensities of fire-protected (FE-APP) and unprotected (FE) laminates is clearly depicted in Figure 2(b) wherein the ΔHRR-time profile is shown. ΔHRR values were obtained by subtracting HRR data measured for FE-APP from that measured for FE (e.g., HRR$_{FE}$ *minus* HRR$_{FE-APP}$). The presence of a char-promoting fire retardant additive within the surface veil significantly reduced the flaming intensity of the flax/epoxy laminates during the initial thermal exposure (e.g., first 200 s) as revealed by positive ΔHRR values over this period, Figure 2(b). A thermally stable and highly consolidated carbonaceous char layer remains at the conclusion of the cone calorimeter test as shown in Figure 3(a). A high degree of surface char consolidation is achieved in this case due to the presence of glass fibers holding the residual material together. Rigid and high char networks serve to inhibit heat transfer into

the laminate and/or the mass transportation of combustible volatiles and oxygen into the pyrolysis zone thereby reducing the flaming-combustion intensity [24]. Being rich in polyhydric compounds (e.g., cellulose, hemicelluloses and lignin), flax fibers should play a crucial role in promoting the generation of polyphosphoric acids a necessary precursor in the formation of a consolidated carbonaceous char [9, 24]. In contrast, only a temporary char would have been formed in FE with that transitory char eventually degrading with prolonged thermal exposure as shown in Figure 3(b). It is noteworthy; despite significant reductions in HRR values, the flame-out time recorded for FE-APP (325 s) is 30% longer than that recorded for the control (250 s). Flame-out times are important fire reaction properties as they influence the total heat release (e.g., integrated area under the HRR-time profiles) during the burning process. It is therefore; not surprising that the THR measured for FE-APP is slightly higher (96 ± 3 MJ/m^2) than that measured for FE (87 ± 2 MJ/m^2).

The average heat rate emission (AHRE) is a vital fire reaction parameter calculated by dividing the cumulative heat release rate by the total exposure time. The maximum average heat rate of emission (MAHRE) is best thought of as an ignition-modified rate of heat emission. This parameter can be used in ranking materials according to their ability to support flame spread to nearby objects. The AHRE-time profiles for both unprotected and fire-protected laminate composites are shown in Figure 4(a). AHRE values calculated for FE are significantly higher than those calculated for the fire-protected laminate (FE-APP) over the duration of the cone calorimeter tests. This observation is clearly revealed when ΔAHRE (AHRE$_{FE}$ *minus* AHRE$_{FE-APP}$) is plotted against exposure time, Figure 4(b). The maximum AHRE calculated for FE (419 ± 8 kW/m^2) is 27% higher than the corresponding valued determined FE-APP (307 ± 15 kW/m^2), Table 1.

The reduction in MAHRE suggests significant alteration in the fire reaction behaviour of the flax/epoxy laminates following the attachment of char-forming fire retardant veil onto the heat-exposed surface. This observation is primarily due to the reduced heat release rates in FE-APP as a result of suppressed flaming-combustion. The presence of a highly consolidated surface char inhibits the conduction of heat and/or

transportation of oxygen into the pyrolysis zone thereby reducing the net heat flux which would have otherwise increased heat emissions. This suggestion is augmented by the delayed mass loss in FE-APP relative to the control laminate, Figure 5(a). While the residual char at the conclusion of cone tests are similar, the average mass loss rate is significantly higher (28%) in FE relative to FE-APP. The effect of surface contained APP on material degradation within flax/epoxy laminates is revealed in the mass difference plot shown in Figure 5(b). At equivalent exposure times, the residual mass for FE-APP is subtracted from that of FE. Negative Δ mass (%) values suggest that material degradation is retarded in the presence of the fire retardant veil at the heat-exposed surface of the flax/epoxy laminate.

The effective heat of combustion (EHC) is another fire reaction parameter that is derived from cone calorimeter data. This parameter which is closely related to the flaming-combustion efficiency is computed by dividing the total heat release by the total mass loss. Effective heat of combustion values were calculated to be 21 and 20 MJ/m^2.kg for FE and FE-APP, respectively. A slightly higher EHC value was calculated for the control laminate and this suggests marginal enhancements in flaming-combustion efficiency of FE. Increased flaming-combustion efficiency in FE relative to FE-APP is augmented by accelerated mass for the former as shown in Figure 5(a). This observation is not unexpected given that carbonaceous char layers have been shown to slow down heat transfer and/or mass transport of combustible volatiles and oxygen into the pyrolysis zone which in turn reduces the flaming intensity of the burning laminate.

Other fire parameters that can be used to rank the fire safety of materials include the fire performance index (FPI) and the fire growth rate (FIGRA). The fire performance index is defined as the ratio of the ignition time to the peak heat release rate (TTI/PHRR). There is a correlation between FPI and the time to flashover; i.e., a lower FPI value suggests an accelerated flashover event. Therefore; materials with low FPI values generally pose elevated fire risks suggesting a higher safety ranking for FE-APP, Table 1. On the other hand, FIGRA values are obtained by dividing the PHRR values by the time required to reach the peak heat release rate

event (PHHR/tPHRR). FIGRA describes the steepest slope of the HRR-time curve. FPI and FIGRA values were calculated for both the control and fire-retarded flax/epoxy laminates, Table 1. The fire growth rate is significantly reduced (12 to 4 $kW/m^2.s$) in the presence of a consolidated char layer at the exposed laminate surface of FE-APP (see Table 1). The reduction in FIGRA is due to the delay in self-sustained ignition and reduced flaming-combustion intensity.

Numerous deaths in building fires are a result of the toxic nature of released fumes. Highly dense smoke may severely reduce visibility slowing down the evacuation process. It is, therefore; vital to understand the effect of fire retardant surface veils on smoke volumes. The total smoke release values were measured as 3038±97 and 3232±73 L for the unprotected and fire-protected flax/epoxy laminates, respectively (see Table 1). Relative to the control laminate, the total smoke release volume measured for the fire-protected laminate is somewhat larger. The marginal increase in the total smoke release for the fire-protected laminate suggests the suppression of the flaming-combustion process in fire-protected laminates. The presence of a physical barrier in the form of a consolidated char layer in FE-APP may slow down mass transportation of combustible volatiles and oxygen into the pyrolysis zone leading to incomplete combustion reactions hence the slight increase in carbon soot/smoke.

3.2 Fire Reaction Properties of Balsa-flax/epoxy Sandwich Composites

Due to the relatively low intrinsic mechanical properties of the bio-reinforcements, the structural/engineering performance of bio-fiber/polymer laminate composites when used alone is usually not adequate [1-3]. Bio-fiber/polymer laminate composites can; however, be incorporated into sandwich composites with the objective of creating environmentally-sustainable and light-weight materials exhibiting improved compression and flexural stiffness. Low-density materials such as wood and polymer foam are usually the materials of choice for the core section of most sandwich panels. However, these materials are highly flammable. That is, the inclusion of inherently flammable bio-fiber/polymer laminates in the designs of

wood- or polymer core sandwich composites can only serve to worsen the fire risks. The fire reaction behaviour of sandwich composites incorporating flammable constituent elements is an under-researched topic. This section of the paper presents an investigation into the fire reaction properties of Balsa core sandwich composites incorporating bio-fiber/epoxy laminate skins. The effectiveness of an innovative fire-proofing approach entailing the bonding a fire retardant glass fiber veil onto the heat-exposed surface of the sandwich composite is evaluated.

The HRR-time data for balsa-flax/epoxy sandwich composites with (BFE-APP) and without (BFE) fire protection are shown in Figure 6(a). Considering the first 400 s of thermal exposure, the reaction of BFE sandwich composites is similar to that observed for flax/epoxy laminate composites (see section 3.1). The HRR of BFE increased rapidly following self-sustained ignition reaching a peak value of 607 ± 22 kW/m^2 after 49 s of continuous thermal exposure. This first peak event in the HRR-time profile was then followed by a rapid decline and then another acceleration in HRR to reach a second peak value of 468 ± 38 kW/m^2 after 237 s. Relative to the HRR values measured for BFE, the flaming intensity of the fire-retarded sandwich composite, BFE-APP, is subdued, Figure 6(a). The first and second peak HRR values for BFE-APP were measured to be 256 ± 15 and 354 ± 11 kW/m^2, respectively. These data represent 58 and 24% reductions in the first and second peak values following the introduction of the char-forming fire retardant at the heat-exposed surface; respectively. Reductions in PHRR values of fire-protected balsa-flax/epoxy sandwich composites are similar to those calculated for fire-retarded flax/epoxy laminate composites. That is, despite the additional fuel from balsa, the flaming-combustion ferocity of balsa-flax/epoxy sandwich composites is not significantly different to that observed in flax/epoxy laminates. However, in spite of similarities in the peak heat release rate values, sandwich composites registered total heat release values three times greater than the values measured for the flax/epoxy laminates. The extended flaming-combustion stage resulting from the additional fuel supplied by the balsa core is implicated for this observation. The reader is reminded that the total heat release is calculated from integrating the area under the HRR-time profile.

The variation in the flaming intensities between the control and the fire-retarded sandwich composites as measured via heat release rates is clearly revealed in the ΔHRR-time data shown in Figure 6(b). Up until 300 s of continuous thermal exposure, the heat release rates measured for the unprotected composite are higher than those measured for BFE-APP. After 400 s of uninterrupted thermal exposure, the sandwich composites (with/without fire protection) begin to smoulder as evident from visual observations and the HRR curves reaching a steady-state value of 100 kW/m^2, Figure 6(a). The HRR-time data suggests that the fire reaction behaviour of flax/epoxy laminate skins is not significantly affected by the presence of highly flammable balsa wood (see section 3.1). Following the thermal degradation of the heat-exposed flax/epoxy skin, (e.g., after 400 s of continuous thermal exposure), flaming-combustion continues to be supported by the balsa core. Eventually, the pyrolysis zone reaches the unexposed flax/epoxy laminate skin on the other side of the balsa-core composite. At this stage, the net heat flux in the system is not sufficient to cause the back skin/laminate to burn with the same ferocity as its heat-exposed counterpart (e.g., front skin). In addition, the charred material from the decomposed heat-exposed skin and balsa core provides a thermal-physical barrier that precludes heat conduction heat and oxygen diffusion to the receding pyrolysis zone. Similar to observations made for detached flax/epoxy laminates, the formation of a consolidated surface char, Figure 7(a), hinders flaming-combustion in BFE-APP sandwich composites. In contrast, no residual char was observed at the heat-exposed surface of BFE sandwich composites, Figure 7(b).

The average heat rate emission profiles were calculated for the unprotected and fire-protected sandwich composites and these data are shown in Figure 8. MAHRE values of 393±13 and 286±9 kW/m^2 were calculated for BFE and BFE-APP, respectively. Interestingly, the percent reduction in calculated MAHRE values for BFE-APP is the same as that calculated for FE-APP. Similarities in MAHRE reduction between flax/epoxy laminates and sandwich composites suggest that the flame retardation mechanism is not altered by the presence of balsa wood in the later. However, the effective heat of combustion values measured for balsa-flax/epoxy sandwich composites are slightly lower than those determined for detached flax/epoxy laminate

composites, Table 1. The reduction in the heat input into the back flax/epoxy skin means that the burning ferocity of sandwich composites decline with uninterrupted thermal exposure. A two-fold increase in the fire performance index was observed following the attachment of APP containing fiber veil at the heat-exposed surface of the sandwich composite, Table 1. The propensity for fire development, otherwise known as FIGRA, is reduced (12 to 4 kW/m^2.s) in the presence of fire retardant veil. The total smoke release values were measured to be 5338±92 and 5617±97 L for BFE and BFE-APP composites, respectively. The presence of a surface char layer during combustion may lead to incomplete combustion process hence the increase in smoke levels in BFE-APP as already discussed in section 3.1.

In summary, the fire retardation strategy employed in this study effectively improved some fire reaction properties of flax/epoxy laminates and balsa-flax/epoxy sandwich composites. Interestingly, the fire retardation efficacy observed in composites incorporating flammable flax reinforcements is similar to that observed for glass fiber-reinforced epoxy composites fire-protected using a char-forming surface coating [19]. Given the increased burn fuel in flax/epoxy laminates and balsa-flax/epoxy sandwich composites, the fire-retarded glass fiber veil performed exceptionally well yielding significant reductions in the flaming intensity and fire growth rates. When percent changes in fire reaction properties are considered, there are no significant differences recorded between flax/epoxy laminates and their balsa-core sandwich composites, Table 1. Similar percent changes were calculated for the PHRR, the THR, MAHRE, the EHC, FIGRA, the FPI and the TSR in both FE-APP and BFE-APP suggesting no significant effect from increased fuel in balsa-flax/epoxy composites.

3.3   Derived Implications on Fire and Post-fire Structural Performance

Research outcomes of this investigation are vital in assessing practical implications of incorporating bio-derived constituent elements on the fire resistance and/or post-fire integrity of structural composites. Specific to engineering structures carrying compressive and/or bending loads, the strength of the laminate face skins

is critical in determining the overall structural performance of sandwich composites. Feih and co-workers reported that sandwich composites simultaneously subjected to compressive and thermal loads fail in a stepped fashion [25]. The initiation and progression of damage leading to catastrophic failure in compression-loaded sandwich composites is largely dictated by the softening temperature of the matrix within the laminate skins. For typical structural epoxy resins, softening of the front skin occurs within the temperature range $60 < T < 150°C$ causing the compressive stress to be transferred to the core and the back skin. Upon failure of the core material, the back skin will continue to carry the applied compressive stress. If the heat supply is uninterrupted, the temperature of the back skin will continue to rise until it reaches the softening temperature. At this point the sandwich composite will fail in a catastrophic manner. Similarly, sandwich composites subjected to flexural loading conditions will progressively lose their structural integrity with continued heat exposure. When the applied bending stress exceeds the flexural strength of the sandwich composite, the structure will fail. Therefore; it is imperative to have fire-proofing methods that can offer enahnced thermal protection for fire-prone engineering structures.

In this paper, temperature-time profiles were collected at locations across the thickness of balsa-flax/epoxy sandwich composites during cone calorimeter tests as illustrated in Figure 1. The temperature-time data at the heat-exposed skin/core interface (T1); balsa core centre (T2) and back skin/core interface (T3) are shown in Figure 9(a) for both BFE and BFE-APP. The temperature at the heat-exposed skin/core interface of BFE increases rapidly to reach a steady-state value of 650°C after 400 s of uninterrupted thermal exposure. The accelerated temperature increase at this location is largely due to cumulative heat release from the flaming-combustion processes following self-sustained ignition. The front skin/core interface temperature stays at 650°C for the entire exposure period due to the incessant incident heat input from the cone heater. Temperature profiles measured at the centre of the core as well as at the back skin/core interface follow the same curvature as that measured at front skin/core interface albeit reaching relatively lower steady-state values; 610 and 490°C, respectively. On the other hand, suppressed flaming-combustion reactions in BFE-

APP led to significantly lower temperature measurements at both the core centre and back skin/core interface. The effectiveness of the fire retardant glass fiber veil in reducing heat conduction into the sandwich composite is clearly revealed in Figure 9(b) wherein the temperature difference between BFE and BFE-APP (e.g., T_{BFE} *minus* $T_{BFE-APP}$) is plotted as a function of exposure time. Temperature difference profiles at the core centre (T2) and the back skin/core interface (T3) are positive over the entire exposure period suggesting that heat conduction across BFE-APP is significantly retarded relative to the control.

When exposed in elevated temperatures, fiber-reinforced polymer composites lose their structural (compressive, tensile or flexural) integrity. The loss in mechanical properties usually occurs at temperature above the glass transition point. The glass transition temperature for typical epoxies is usually between 100 and 150°C. From previous studies, glass fiber/epoxy laminates lost all their ambient temperature mechanical strength and stiffness at temperatures above 150°C [18]. In this study, the time taken by the back face laminate to reach a temperature of 150°C was recorded for BFE and BFE-APP sandwich composites. Relative to BFE, a 250 s delay in the back skin temperature of BFE-APP reaching 150°C was observed. Catastrophic collapse is triggered when the stressed back skin laminate achieve a temperature of 150°C. In the case of BFE-APP, building occupants will have an additional 4 mins before structural collapse.

An additional and equally important parameter in the design of fiber/polymer composite structures for deployment in fire-prone environments is the onset temperature of decomposition. The loss of combustible materials (e.g., thermal decomposition of the organic matrix and/or degradation of the fiber reinforcements) has adverse effects on the structural integrity of composites during and after fire exposure [25-31]. The onset temperature of decomposition for epoxies and flax fiber is usually between 300 and 350°C [18, 32]. In this paper, a decomposition temperature of 300°C was chosen as a reference point to afford a comparative analysis between fire-protected and unprotected balsa-flax/epoxy sandwich composites. When compared to the fire-retarded composite, the onset temperature of decomposition at the back skin/core interface of the

control is reached 360 s earlier. It should be noted; however, the delay in the onset of thermal decomposition for the epoxy matrix or bio-fiber may have no physical significance to structural resilience of fiber/polymer composite elements during fire. This is due to the fact that softening temperatures precede the onset of thermal decomposition. The global structure could have already collapsed before the onset temperature of decomposition is reached depending on the loading scenario. However, if the fire is extinguished before the structure collapses, it is crucial to have an idea of the extent of damage and residual strength [18, 28].

4.0   CONCLUSIONS

The fire retardation efficacy of ammonium phosphate-carrying glass fiber veil was evaluated using the cone calorimeter. The effectiveness of the fire retardant veil was evaluated for highly flammable composites incorporating bio-derived constituents under thermal conditions simulating a fully-developed room fire.   The presence of ammonium phosphate and a glass veil at the heat-exposed surface of evaluated composites promoted the formation of a highly consolidated physical and thermal barrier. The existence of this rigid carbonaceous char served to reduce heat conduction and/or mass transport of combustible volatiles and oxygen into the pyrolysis zone thereby reducing the flaming-combustion intensity. Fire-retarded bio-fiber/epoxy laminates and their corresponding balsa-core sandwich composites showed improved fire reaction properties. The heat release rates at equivalent thermal exposure times were significantly lower for the fire-retarded flax/epoxy laminate and balsa-core sandwich composites when compared to their unprotected counterparts. Measured and derived cone calorimeter data suggest that the presence of a fire retardant glass fiber veil effectively contained fire spread. The reduction in fire growth rates and the depleted propensity to cause fire in fire-retarded flax/epoxy laminate and balsa-core sandwich composites is notable. Regardless of the significantly increased burn fuel, the fire retardation strategy considered was adequate in significantly increasing the fire resistance and post-fire structural resilience of the concerned composites. The improvements realized in fire reaction behaviours of fire-protected balsa-flax/epoxy sandwich composites may be sufficient to allow their deployment in semi-structural engineering applications threatened by fire.

Acknowledgements

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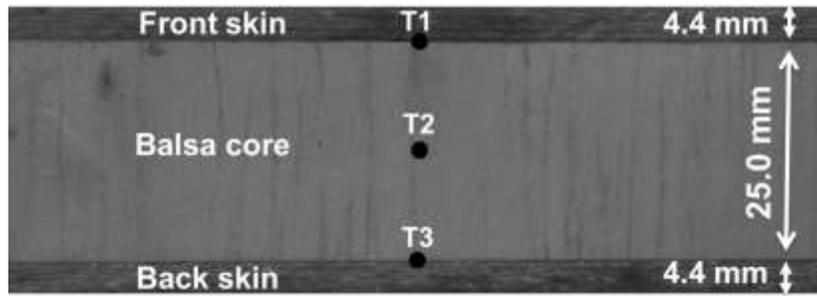
Figure 1. An illustration showing the location of thermocouples inserted across the thickness of some balsa-flax/epoxy sandwich composites during cone calorimeter tests.
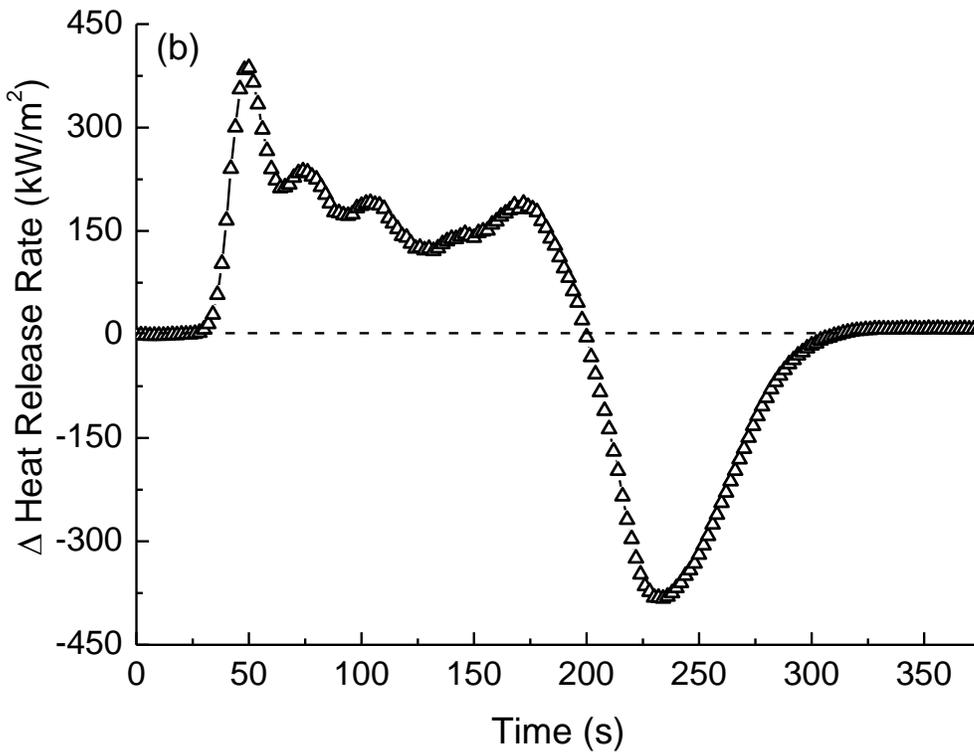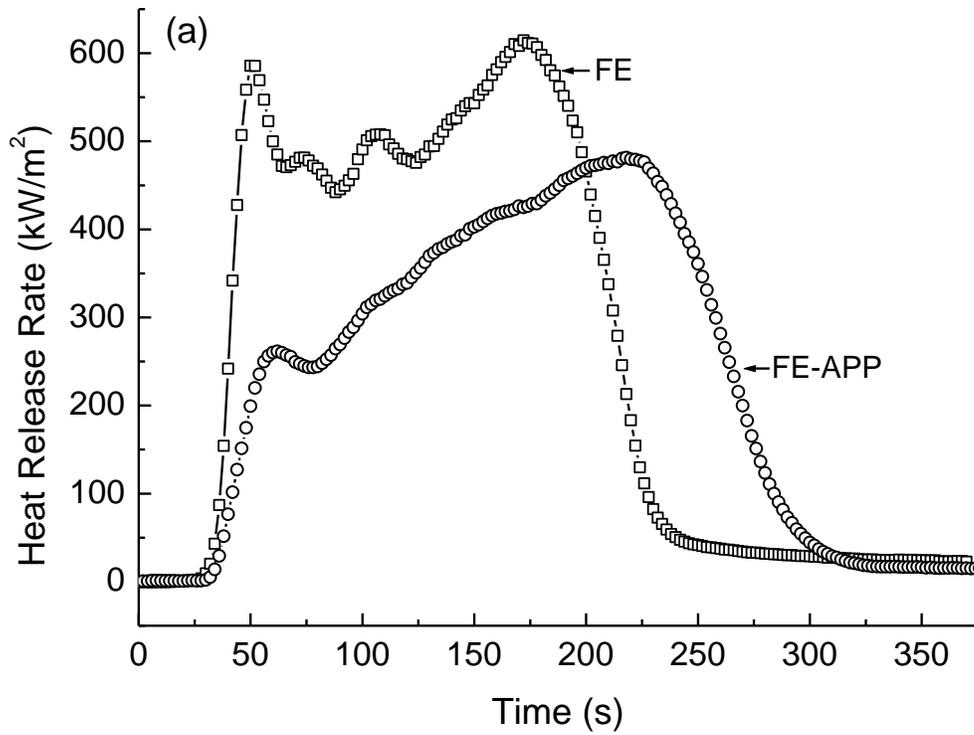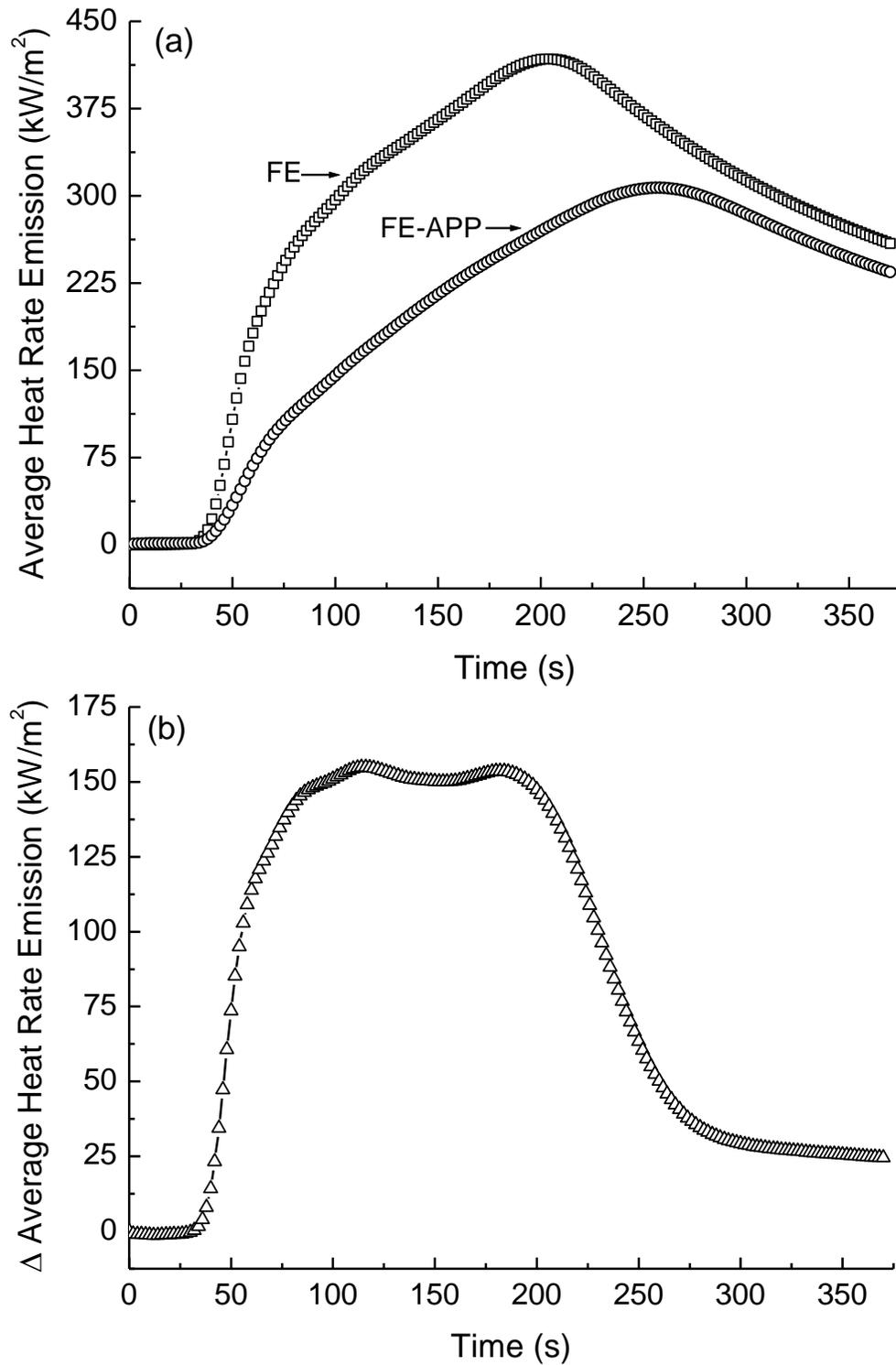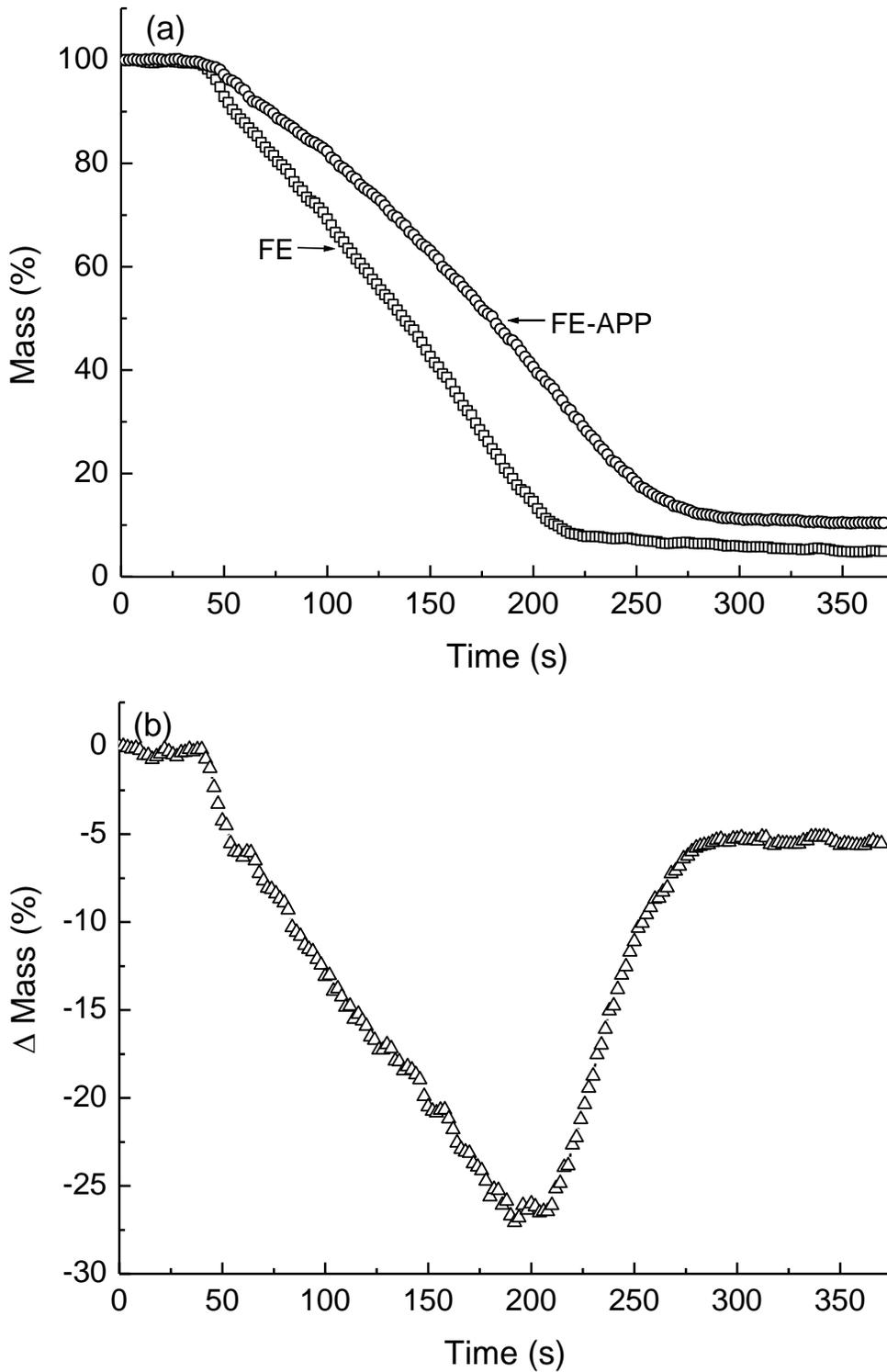
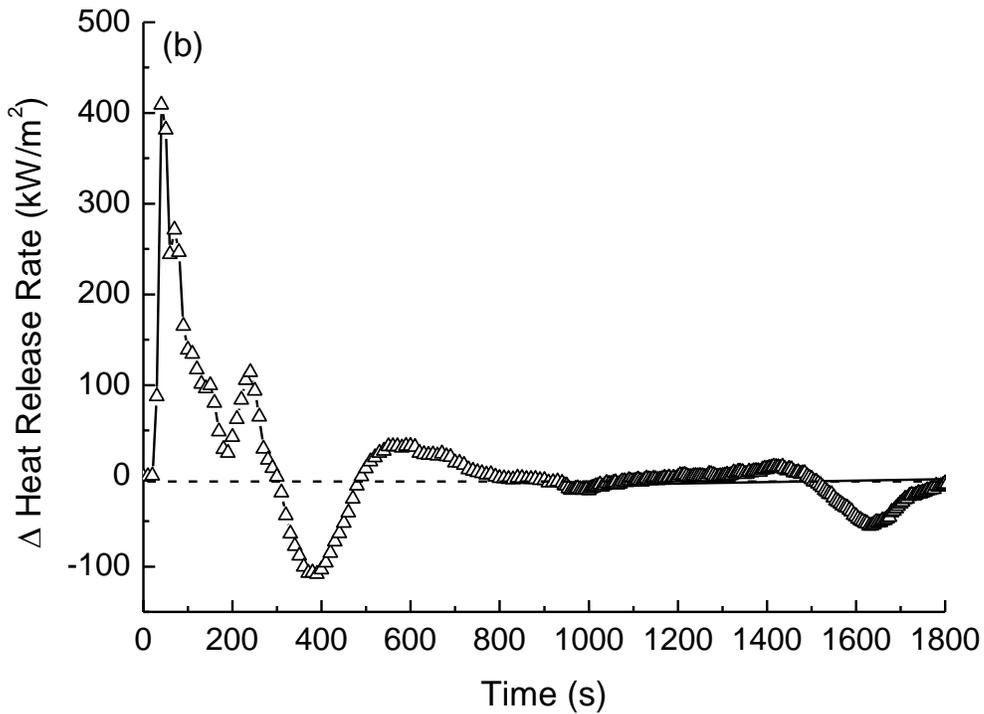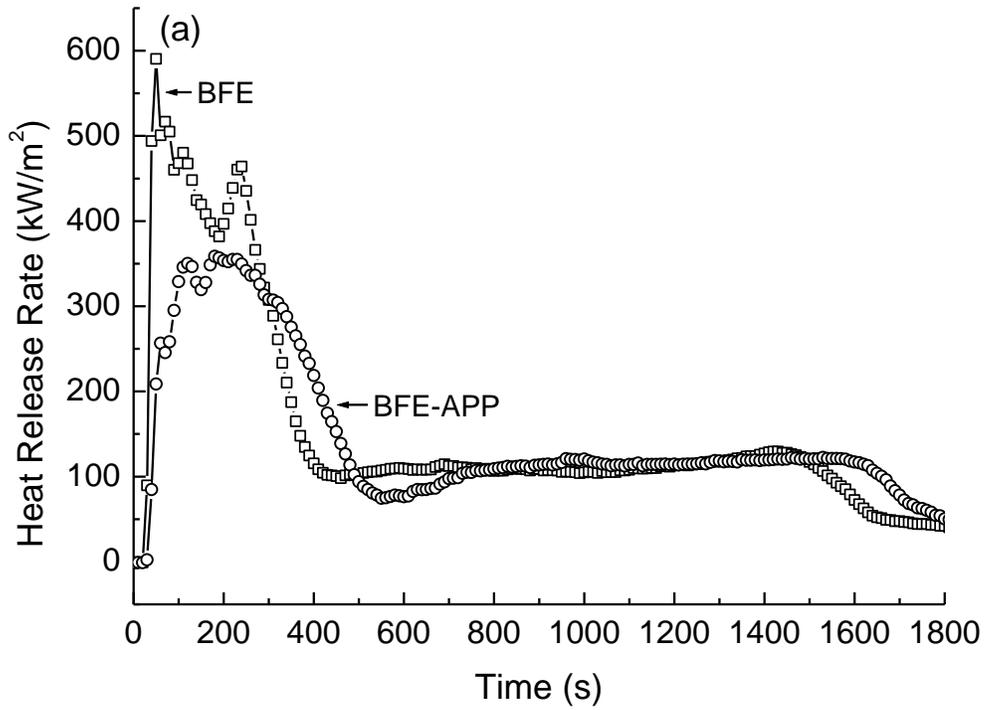Figure 2. Heat release rate data for flax/epoxy laminates with (FE-APP) and without (FE) fire protection exposed at an applied heat flux of 50 kW/m^2; (a) measured heat release rates and (b) calculated Δ heat release rate, respectively.
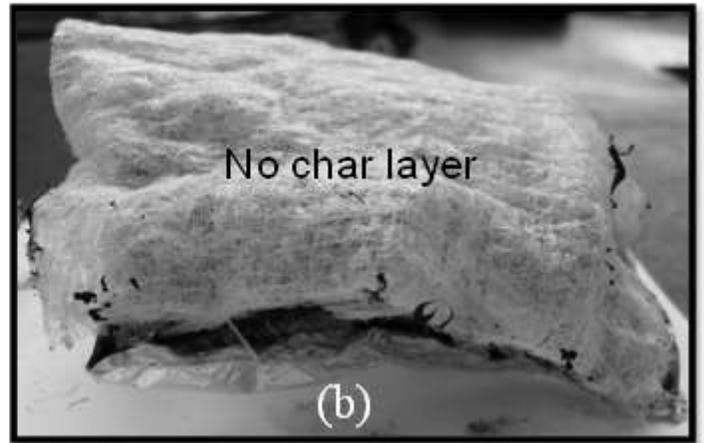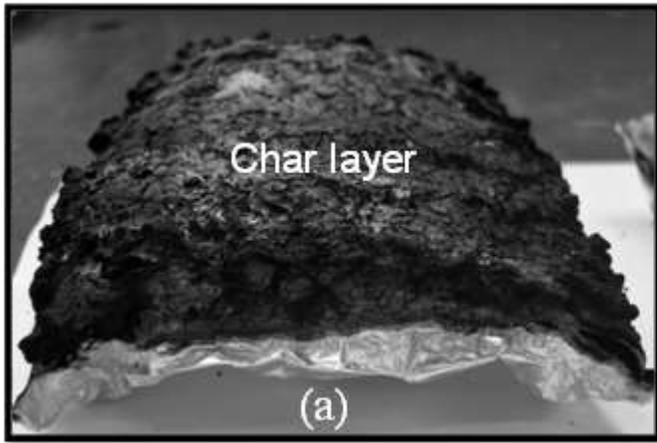
Figure 3. Photographic images of fire-damaged flax/epoxy laminates; (a) with (FE-APP) and (b) without (FE) fire protection, respectively.

Figure 4. Average heat rate emission data for flax/epoxy laminates with (FE-APP) and without (FE) fire protection at an applied heat flux of 50 kW/m^2; (a) measured average heat rate emission and (b) calculated Δ average heat rate emission, respectively.

Figure 5. Mass data for flax/epoxy laminates with (FE-APP) and without (FE) fire protection at an applied heat flux of 50 kW/m^2; (a) measured mass and (b) calculated Δ mass, respectively.

Figure 6. The heat release rate data for balsa-flax/epoxy sandwich composites with (BFE-APP) and without (BFE) fire protection at an applied heat flux of 50 kW/m^2; (a) measured heat release rate and (b) calculated Δ heat release rate, respectively.
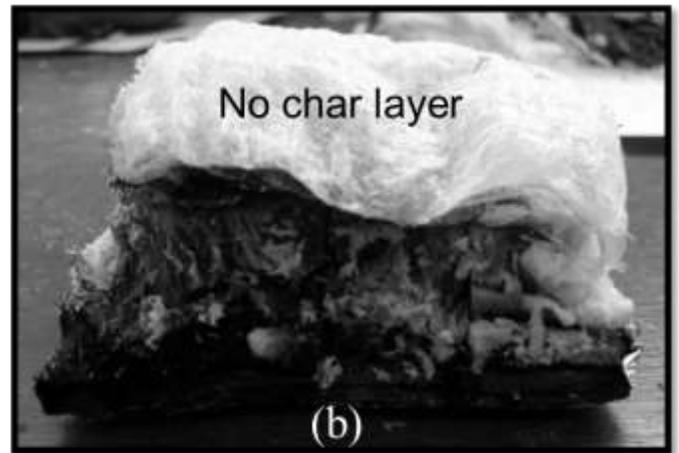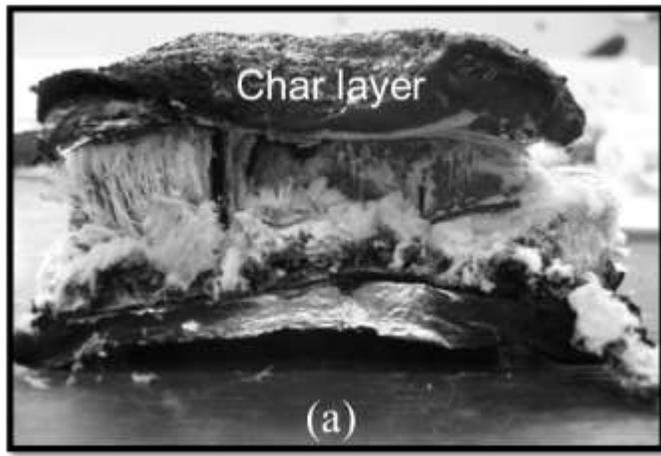
Figure 7. Photographic images of fire-damaged balsa-flax/epoxy sandwich composites; (a) with (BFE-APP) and (b) without (BFE) fire protection, respectively
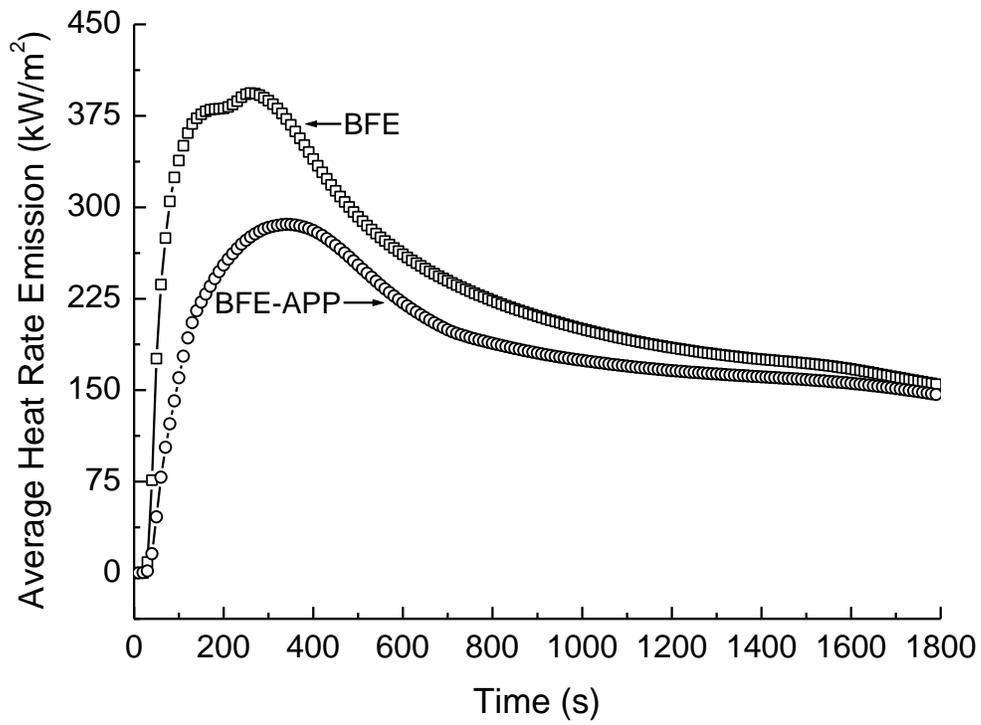
Figure 8. Average heat rate emission data for balsa-flax/epoxy sandwich composites with (BFE-APP) and without (BFE) fire protection at an applied heat flux of 50 kW/m^2.
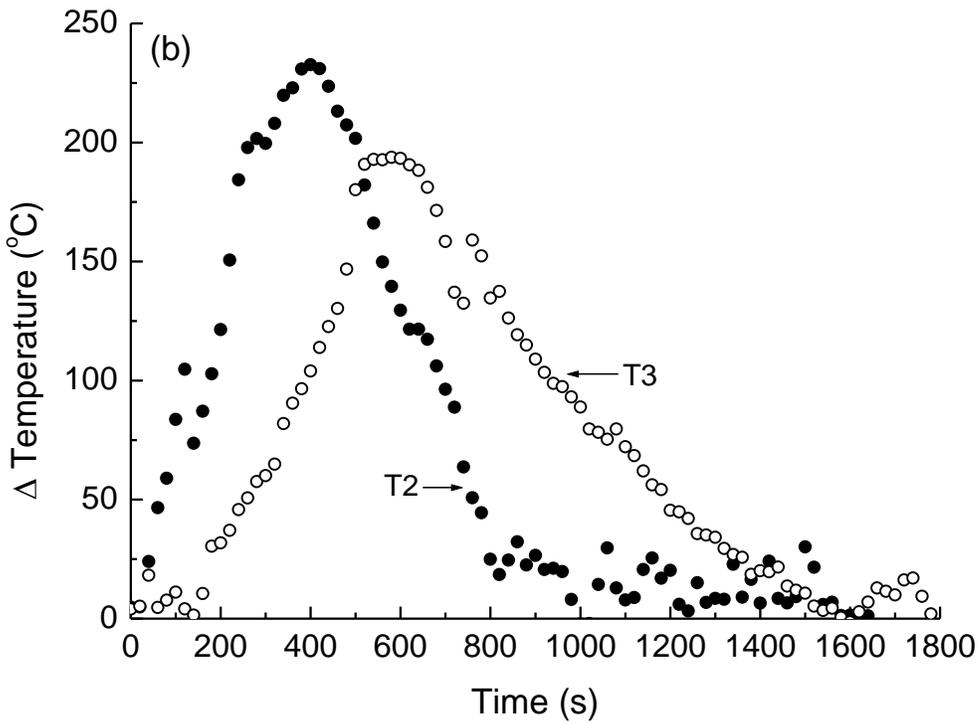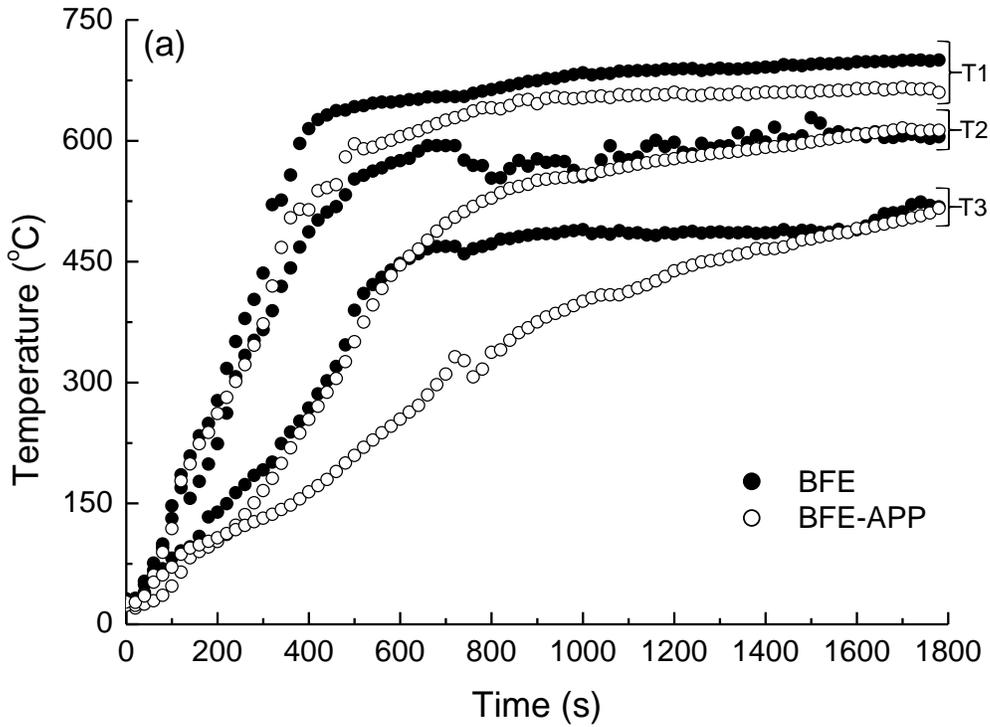
Figure 9. Temperature data for balsa-flax/epoxy sandwich composites with (BFE-APP) and without (BFE) fire protection at an applied heat flux of 50 kW/m^2; (a) measured temperature-time data and (b) calculated Δ temperature values at two locations, T2 and T3, respectively.

Table 1: Cone calorimeter data measured for flax/epoxy laminate and balsa-flax/epoxy sandwich composites (with/without fire protection) subjected to an applied heat flux of 50 kW/m^2 in the presence of an ignition source.

Sample	TTI (s)	PHRR[1] (kW/m^2)	tPHRR[1] (s)	PHRR[2] (kW/m^2)	tPHRR[2] (s)	THR (MJ/m^2)	MAHRE (kW/m^2)	EHC (MJ/m^2.kg)	FIGRA (kW/m^2.s)	FPI (s.m^2/kW)	TSR (L)
FE	35	592	51	616	173	96	419	21	12	0.06	3038
FE-APP	32	262[−56]	62[+22]	485[−21]	211[+22]	87[−9]	307[−27]	20[−6]	4[−67]	0.12	3232
BFE	29	607	49	468	237	278	393	19	12	0.05	5338
BFE-APP	30	256[−58]	61[+24]	354[−24]	234[−1]	263[−5]	286[−27]	17[−10]	4[−67]	0.12	5617

TTI is the time-to-ignition; PHRR is the peak heat release rate; tPHRR is the time to reach the peak value of the HRR; THR is the total heat release; MAHRE is the maximum average heat rate emission; EHC is the effective heat of combustion; FIGRA is the fire growth rate calculated from PHRR/tPHRR[1]; FPI is the fire performance index calculated from TTI/PHRR[1] and TSR is the total smoke release. The numerical symbols [1] and [2] refer to the first and second HRR peak in HRR-time profiles, respectively. The percent changes in fire reaction parameters following the introduction of the fire retardant veil are given in square brackets [] with the (−) and (+) signs representing reductions and enhancements, respectively.