Bhat T, Kandare E, Gibson AG, DiModica P, Mouritz AP.
[Tensile properties of plant fibre-polymer composites in fire.](#)
***Fire and Materials* 2017,**
<https://doi.org/10.1002/fam.2446>

Copyright:

This is the peer reviewed version of the following article: Bhat T, Kandare E, Gibson AG, DiModica P, Mouritz AP. [Tensile properties of plant fibre-polymer composites in fire.](#) *Fire and Materials* 2017, which has been published in final form at <https://doi.org/10.1002/fam.2446>. This article may be used for non-commercial purposes in accordance with Wiley Terms and Conditions for Self-Archiving.

Date deposited:

02/05/2017

Embargo release date:

13 June 2018

TENSILE PROPERTIES OF PLANT FIBRE-POLYMER COMPOSITES IN FIRE

T. Bhat^a, E. Kandare^a, A.G. Gibson^b, P. Di Modica^b, A.P. Mouritz^a

^a Sir Lawrence Wackett Aerospace Research Centre, School of Engineering, RMIT University, Melbourne, Victoria 3000, Australia

^b Centre of Composite Materials and Engineering, University of Newcastle-upon-Tyne NE1 7RU, UK

Corresponding author:

Adrian P. Mouritz

Tel: +61 3 99256269

Fax: +61 3 99256108

e-mail: adrian.mouritz@rmit.edu.au

ABSTRACT

The structural performance of polymer composites reinforced with plant fibres when exposed to fire is experimentally evaluated, and compared against an E-glass fibre laminate. Fire testing under combined one-sided radiant heating and static tensile loading revealed that flax, jute or hemp fibre composites experience more rapid thermal softening and fail within much shorter times than the fibreglass laminate, which is indicative of vastly inferior structural performance in fire. The plant fibre composites soften and fail before the onset of thermal decomposition of the plant fibres and polymer matrix, whereas the E-glass fibres provide the composite with superior tensile properties to higher temperatures and higher applied tensile stresses. The tensile performance of the three types of plant fibre composites in fire were not identical. When exposed to the same radiant heat flux, the flax fibre composite could withstand higher tensile stresses for longer times than the hemp and jute laminates, which showed similar performance.

Keywords: Natural fibre composite, fire, thermal properties, mechanical properties

1 INTRODUCTION

There has been a large increase in the use of fibres extracted from plants as a substitute to synthetic fibres such as glass and carbon for the reinforcement of polymer matrix composites. Carus and Scholz [1] predict that bio-fibres will account for ~28% of the total composite reinforcements by the year 2020. The most common plant fibres used in composites are flax, jute and hemp [2]. The key incentive towards the use of plant fibres is moderate cost, low environmental impact and carbon footprint, biodegradability, light-weight, and good specific mechanical properties. Although the mechanical properties of plant fibres are not as high as glass and carbon fibres, the controlled processing and surface treatment of plant fibres has allowed their use in composites for semi-structural applications [3-6]. For example, there is interest to use natural fibres in load-bearing composites in automobiles and construction [7-9].

The mechanical properties of plant fibres and their polymer composites have been studied extensively [9-18]. However, much less is known about their fire reaction and fire resistance (including structural integrity) properties [19-24]. Plant fibre composites potentially pose an increased fire risk when used in load-bearing structures compared to glass and carbon fibre composites due to the higher organic (fuel) content [20]. Plant fibres may shorten the ignition time and lower the flaming combustion temperature, increase the heat release rate, generate thicker smoke, and increase other fire risk factors such as flame spread and smoke toxicity. For these reasons, plant fibre composites often contain flame retardant additives within the polymer matrix 25. For example, Subasinghe and Bhattacharyya 26 blended one of three types of ammonium polyphosphate (APP) in the polypropylene matrix of kenaf bast fibre composites, and found an improvement to the flame retardant properties. Another potential fire hazard is that plant fibres weaken and fail at lower temperatures and within shorter times than glass or carbon fibre composites when exposed to fire. It is therefore expected that plant fibre composites will have inferior structural performance in fire, although this is yet to be

quantitatively proven. Just how much the structural properties of plant fibre composites are reduced in fire is not known. Also, it is not known whether any differences exist between different types of plant fibres used in composite materials.

In this paper, fire structural performance is defined as the capacity of a material to retain load-bearing properties when exposed to fire. Understanding the structural performance in fire of any new material, including plant fibre composites, with potential applications in load-bearing structures is critical because the loss in strength and stiffness can cause failure and collapse, resulting in human fatalities and/or property losses. Plant fibres experience almost complete loss in strength by $\sim 200^{\circ}\text{C}$ [28] and begin to thermally decompose at $\sim 250^{\circ}\text{C}$ [16,21], with this value varying slightly depending upon the chemical composition of the specific fibre [25]. Plant fibres begin to lose tensile strength at much lower temperatures compared to E-glass fibres (above $\sim 350\text{--}400^{\circ}\text{C}$) [29] and carbon fibres (above $\sim 500^{\circ}\text{C}$) [30]. It is likely that plant fibre composites are structurally inferior to glass or carbon fibre composites when exposed to fire, although by how much remains unclear because their fire structural performance have not been evaluated.

The impact of substituting E-glass fibres with plant fibres on the structural properties of polymer matrix composites in fire is experimentally evaluated in this paper. The tensile performance in fire of composites reinforced with flax (*Linum usitatissimum*), jute (*Corchorus capsularis*) or hemp (*Cannabis sativa L*) fibres are determined, and benchmarked against an E-glass laminate with the same fibre volume content and type of polymer matrix. The composites studied here do not contain flame retardant additives or other materials (e.g. intumescent coatings) to enhance their performance in fire, even though they are often used in plant fibre laminates for commercial applications. This is to allow the single effect of the influence of the plant fibre type on the tensile performance of the laminate in fire to be quantified by testing. The structural performance in fire of the flax, jute, hemp and glass fibre composites was experimentally evaluated using small-scale fire tests involving combined static tensile loading and one-sided

heating at a constant heat flux. This test mimics the condition of a tensile load-bearing beam subject to fire attack. In addition, elevated temperature tensile tests were performed on plant fibre tows as well as their polymer composites to identify the softening mechanisms controlling the strength loss when exposed to fire. The results from this study provide the first quantitative data into the impact of replacing E-glass fibres with plant fibres on the fire structural safety of polymer composites.

2 MATERIALS AND EXPERIMENTAL METHODS

2.1 Composite Materials

Vinyl ester matrix composites reinforced with the same volume content ($40 \pm 2\%$) of plant fibres (jute, hemp or flax) or E-glass fibres were produced. The jute, hemp and flax were cleaned by the supplier. The jute was supplied by Composites Evolution Ltd in the form of plain woven fabric (areal density of 290 g/m^2) with a 50:50 ratio of warp [0°] and weft [90°] 250 tex twisted tows. The hemp was also in the form of a plain woven fabric (287 g/m^2) with an equal ratio of warp and weft twisted tows with 250 tex, and was supplied by Hemp Wholesale Australia. The jute and hemp fabrics were stacked in multiple layers with the warp-weft tows always aligned in the same directions. This resulted in a cross-ply fibre architecture consisting of all of the warp tows being aligned in the same direction (0°) and all of the weft tows aligned in the normal direction (90°) in the composite material. The flax was in the form of unidirectional fabric (180 g/m^2) containing 42 tex twisted tows supplied by SAS Lineo. The unidirectional flax plies were stacked in a cross-ply [$0^\circ/90^\circ$] sequence to replicate the 50:50 ratio of warp and weft tows in the jute and hemp fabrics. That is, the unidirectional fabrics were stacked in an alternating pattern of 0° and 90° in the through-thickness direction to create a cross-ply fibre pattern. Provided in Table 1 is the approximate composition of the different plant fibres. Sizing agent was not present on any the plant fibres. The E-glass was a plain woven fabric (areal density = 830 g/m^2)

containing sized 300 tex tows, and supplied by Colan Australia. The glass fabric contained an equal ratio of warp and weft yarns.

The plant fibres were infused with vinyl ester resin (SPV 1265 supplied by Nuplex Composites) at room temperature using the vacuum bag resin infusion (VBRI) process. Following the infusion, the composites underwent gelation and partial curing under ambient conditions ($23 \pm 2^\circ\text{C}$, $50 \pm 5\%$ RH) over several hours, and were then post-cured at 80°C for two hours. The fibre volume content of all the plant composites was measured to be $40 \pm 2\%$ using ASTM D-3171 31. In order to match the fibre volume content of the plant fibre composites, the fibreglass composite was made using the wet hand lay-up process. This composite was gelled and cured under the same conditions as the plant fibre materials. The plant and glass fibre composites were made to two thicknesses: 4 mm for high temperature property testing and 9 mm for fire-under-load and cone calorimetry testing. The glass transition temperature (T_g) of the vinyl ester matrix following post-curing was 110°C for the different composites, which was measured using DMTA as the 50% reduction in the storage modulus compared to the room temperature value.

2.2 High Temperature Testing of Fibre Tows and Composites

2.2.1 Thermal-Mechanical Testing of Fibre Tows

The tensile properties of jute, hemp, flax and E-glass fibre tows were measured at temperatures between 20°C and 250°C using the procedure illustrated in Figure 1. The tows were carefully extracted from the same fabric used to fabricate the composites, making sure that no damage was caused to the fibres. The tow ends were wound around circular rollers (150 mm diameter) leaving a gauge section of 100 mm long. A 10 mm middle section of the tow was then heated to the test temperature using a hot air gun fitted with a variable temperature controller. The temperature was measured using a K-type thermocouple in contact with the tow. The fibre tow was heated for a soak period of two minutes prior to tensile loading to ensure that all the fibres were at the test temperature. The tow was then loaded to failure at a displacement rate of 2

mm/min using a 10 kN Instron machine (Model 5569) while at elevated temperature. Five tows of the same fibre type were tested at each of the temperatures. The average failure loads for flax, jute and hemp tows were respectively 15 N, 28 N and 43 N, while for the E-glass tows it was 125 N at 20°C.

2.2.2 Thermal-Mechanical Testing of Composites

The tensile modulus and failure stress of the plant and glass fibre composites were measured at different temperatures (20°C - 200°C) inside an oven attached to a 50 kN Instron test machine. The tensile tests were performed in accordance with ASTM D-638 32 using a dog-bone shaped coupon with a gauge length of 57 mm, width of 13 mm and thickness of 4 mm. The tensile tests were performed under isothermal conditions by loading the composites in the warp (0°) fibre direction at a displacement rate of 2 mm/min until failure. Three samples of each type of composite were tested at the different temperatures.

2.2.3 Fire-Under-Load Testing of Composites

An experimental comparative analysis of the fire resistant properties of the plant and glass fibre composites were performed using data obtained from small-scale fire-under-load tests. The design of the test was to replicate a tensile-loaded flat beam subjected to one-sided heat radiated from a fire. A schematic representation of the test is illustrated in Figure 2. It is important to note that no standardised test method exists to determine the fire structural performance of composites or other materials subjected to combined mechanical loading and one-sided heating representative of fire. While many test methods exist, including using different methods to replicate the fire such as infrared-red strip heaters 33,34], gas flame burners 35,36 and furnaces 37,38 among other techniques 39-42. Unfortunately, there is no single method which is universally used and standardised. For this reason, a customised method which has been used in other studies 43 to determine the mechanical response of structural materials (including

composites) exposed to simulated fire was applied here to comparatively assess the fire performance of the plant and glass fibre composites.

The composite specimens were 600 mm long, 50 mm wide and ~9 mm thick, and were loaded in tension to a constant stress between 20% and 80% of the room temperature failure strength using a 250 kN MTS machine. The tensile load was applied parallel to the warp (0°) fibre direction. While under constant stress, the specimen was subjected to one-sided heating using a 5000W cone shaped radiant heater. The incident heat flux radiated from the cone heater was measured before testing using a heat flux gauge (Model no. 32-10SB-10-197-21633) supplied by Hukseflux Thermal Sensors. Also, the cone heater was fitted with a thermocouple which was connected to a thermal control system which continuously adjusted the power supplied to the radiant heater to ensure a constant heat flux. Using this controller system, the different composites were fire tested under identical conditions.

A 100 mm long x 50 mm wide section of the composite was exposed directly to the heater located 25 mm away, while the rest of the specimen was thermally insulated using a 10 mm thick FibreFrax ceramic blanket supplied by Unifrax Australia Pty Ltd. Fire-under-load tests were performed at incident heat fluxes of 10 kW/m² and 35 kW/m², which were selected because they represent heating conditions for two fire scenarios that induce different types of thermal softening. The lower heat flux of 10 kW/m² heated the composites to above the glass transition temperature of the polymer matrix (110°C), but the flux was too low to cause significant decomposition. The higher heat flux of 35 kW/m² resulted in higher temperatures that caused softening and decomposition of the polymer matrix and plant fibres. During fire structural testing the composites were subject to simultaneous tensile loading and one-sided heating until total failure.

2.2.4 Fire Reaction Testing of Composites

The fire reaction properties of the plant and glass fibre composites were measured using a three-cell cone calorimeter (Model no: 5023431 by Fire Testing Technology Ltd) operated in the vertical testing mode. Test specimens (100 mm long $\times$ 100 mm wide $\times$ 9 mm thick) were exposed to an incident heat flux of 10 or 35 kW/m² (without a spark ignitor) according to the ISO 5660 standard 46. The heat-exposed surface of the specimen was positioned 25 mm from the cone heater. The mass loss, heat release and smoke density properties were measured at the two heat flux values. The heating conditions to measure the fire reaction properties using the cone calorimeter were very similar to the conditions experienced by the composites during the fire-under-load tests.

3 RESULTS AND DISCUSSION

3.1 High Temperature Properties of Plant and Glass Fibre Tows

Thermal softening of the fibre tows has a direct influence on the tensile performance of composites exposed to fire. For this reason, tensile tests were performed on the flax, jute, hemp and E-glass tows up to 250°C, which is just below the onset temperature for thermal decomposition of plant fibres [16,47,48]. The effect of increasing temperature on the tensile stiffness and failure load of the different tows are shown in Figure 3. The tensile properties are normalised to the tex value of the tow, which is different for the different fibre types. As expected, the tensile properties of the E-glass tow was higher than for the plant fibre tows. Plant fibre tows consist of twisted and discontinuous filaments while glass fibre tows consist of continuous filaments. The higher twist, shorter length, intrinsically lower strength, higher flaw contents, filament shape, and poor cohesive forces between the fibres are among the reasons for the plant fibres being weaker. It is shown in Figure 3 that the glass fibre tow retained both its tensile strength and stiffness up to 250°C. Feih et al. 49 have shown that the failure stress of E-glass fibres and tows does not begin to decrease until the temperature exceeds ~250°C. In contrast, the tensile properties of the plant tows decreased at a quasi-linear rate between 20°C

and 250°C, with the jute and hemp softening at similar rates. The flax retained higher stiffness and strength than the jute and hemp over the range of temperatures.

Many studies have measured the reductions to the failure stress of plant fibres at elevated temperatures 28,48,50-53 , and similar trends have been reported to those shown in Figure 3. However, the tensile properties of fibre tows may differ from their respective individual filaments at room and elevated temperatures due to the influences of fibre architecture (e.g. twist angle, fibril lengths, etc). Thermal weakening of plant fibres is generally attributed to evaporation of water; glassy-to-rubbery type softening of crystalline cellulose and lignin; and physical damage. Plant fibres derive significant strength from crystalline cellulose fibrils, which are bundled into mesofibrils 54. The mesofibrils are embedded in an amorphous polysaccharide matrix composed mostly of pectin and hemicellulose 51. At ~100°C, the physical desorption of water embrittles the plant fibres, especially via dehydration of the gel-like network formed by the polysaccharides 50. Water loss reduces the stress transfer efficiency between the load-bearing mesofibrils and the pectin matrix, thereby reducing fibre strength. Lignin and cellulose undergo glassy-to-rubbery type transition softening at temperatures of ~125°C and 200°C, respectively, which further weakens plant fibres 51. Flax generally has a lower lignin content than jute and hemp [21], and this in part may account for its higher stiffness and strength as shown in Figure 3. The failure stress is also reduced by cracking within the fibres caused by differences between the thermal expansion coefficients of cellulose, hemicellulose and lignin 52. Flaws initiate at the interfaces between these constituents, which grow with increasing temperature and develop into cracks under tensile loading which lower the failure stress [52,53.

3.2 High Temperature Properties of Plant and Glass Fibre Composites

The thermal softening behaviour of plant and glass fibre composites was investigated by conducting tensile tests at different temperatures up to 200°C. As shown in Figure 4, the tensile strength of the glass fibre composite (440 MPa) was much higher than the plant fibre laminates

at 20°C due to their lower fibre failure stress and the lack of sizing agent. The tensile strength of the flax fibre composite (150 MPa) was higher than the jute or hemp laminates (both ~50 MPa) at 20°C, due to the higher failure stress of flax tows [17,47,48]. Also, the flax fibres were used in the form of unidirectional cloth whereas the jute and hemp were in the form of plain woven fabric. The weaving process can cause damage to the fibres, and this may have contributed to the lower failure stress values of the jute and hemp composite materials at room temperature.

The tensile properties of the glass fibre composite decreased with increasing temperature due to softening of the polymer matrix. Since glass fibres do not lose stiffness and strength below 200°C, the reduction to the tensile properties of the glass fibre composite was due to glass transition softening of the matrix and the resultant loss in stress transfer efficiency between the load-bearing fibres. The plant fibre composites softened progressively with increasing temperature, with little load-bearing capacity at the maximum temperature of 200°C. The losses in stiffness and strength for these composites was due to a combination of glass transition softening of the polymer matrix and thermal softening and damage to the plant fibres (as described above).

3.3 Tensile Performance of Plant and Glass Fibre Composites in Fire

3.3.1 Low Temperature Fire Exposure (Heat Flux = 10 kW/m²)

The fire-under-load properties of the plant fibre composites are compared to the fibreglass laminate when exposed to the incident heat flux of 10 kW/m². As mentioned, this flux was chosen because it is high enough to cause softening of the composites but too low to cause any significant decomposition to the polymer matrix or plant fibres or weakening of the glass fibres, and therefore represents a low temperature fire scenario. This is evident from the heat release rate curves measured using cone calorimetry for the different composites at the heat flux of 10 kW/m², as shown in Figure 5. The different composites did not release any heat with increasing exposure time to the heat flux, which is evidence that none of the materials decomposed. The

small fluctuations in the heat release rate values are due to the experimental noise in the measurement of heat release rate, and not due to heat being released by the composites.

The temperatures of the flax, jute, hemp and E-glass fibre composites when exposed to the heat flux of 10 kW/m^2 for increasing time is shown in Figure 6. Temperature-time histories were measured using thermocouples located at the front (heat-exposed) and back surfaces of the composites. Multiple tests performed on the same composite revealed little variability in the measured temperature (typically within 15°C), and therefore any temperature differences greater than this between the materials is significant. It is shown in Figure 6 that exposing the plant and glass fibre composites to the same heat flux caused an initial rapid rise in the front surface temperature up to the maximum steady-state value of $\sim 220^\circ\text{C}$ that was reached after ~ 30 mins. The maximum temperature of the front surface was above the glass transition temperature (110°C) but below the decomposition temperature ($\sim 350^\circ\text{C}$) of the vinyl ester matrix. Likewise, the maximum temperature was above the softening temperature but below the decomposition temperature of the plant fibres. It is for this reason that cone calorimetry failed to detect any measurable increase in the heat release rate of the composites when exposed to the heat flux (Figure 5).

The temperatures decreased from the front to the back surface of the plant and glass fibre composites. The back surface temperatures of the plant fibre composites were similar, and lower than the glass fibre laminate. This suggests the plant fibre composites have superior insulating properties when exposed to the low heat flux. The plant and glass fibres have similar thermal conductivity values (typically around $0.038\text{-}0.04 \text{ W/m K}$) [45], which suggests their composites should also have similar heat insulation properties (provided the fibre content and orientations are the same). However, the cooler internal and back surface temperatures of the plant fibre composites was due to restricted heat flow caused by heat-induced damage and moisture evaporation. For example, Figure 7 shows that the amount of delamination cracking in the plant

fibre composites was greater than the glass fibre laminate. Also, fibre-matrix interfacial cracks occurred in the plant fibre composites which did not occur in the glass fibre material. The higher amount of delamination and interfacial cracking is attributed in part to the absence of sizing agent on the plant fibres. Weakening/embrittlement of the plant-matrix interface caused by evaporation of water may have contributed to interfacial cracking, which increased the apparent thermal insulation properties of the plant fibre composites.

Delamination and fibre-matrix interfacial cracks slow heat flow in the through-thickness direction, and this would account in part for the lower back surface temperatures of the plant fibre composites. It is also possible that the evaporation of water from the natural fibre composites contributed to the lower back surface temperatures. Mass loss-time curves measured for the composites when exposed to the heat flux of 10 kW/m^2 in the cone calorimeter are shown in Figure 8. The plant fibre composites showed an almost immediate and progressive reduction in mass upon exposure to the heat flux, and this was due to the evaporation of water from the fibres. In comparison, the glass fibre laminate did not experience any mass loss. The transport of hot water vapour through the plant fibre composites and its evaporation from the front surface has an internal cooling effect, and this may have contributed to the lower temperatures.

The effect of increasing exposure time to the heat flux of 10 kW/m^2 on the tensile failure stress of the different composites is shown in Figure 9. As expected, the failure stress decreased rapidly with increasing heat flux exposure time, and similar trends have been reported for glass fibre and carbon fibre laminates [30,33-36,43-45]. The failure stress values of the plant fibre composites were much lower than the fibreglass laminate, even though the heat flux was too low to cause significant thermal decomposition of their fibres. The fire structural performance of the flax fibre composite was slightly better than the jute and hemp laminates (which were similar), and this correlates with the differences in the failure stress of both the tows and composites at elevated temperature (Figs. 3 and 4).

The rapid deterioration to the tensile performance of the plant fibre composites when exposed to the heat flux was due to thermal softening of both the polymer matrix and fibre reinforcement at moderately low temperatures (under 200-250°C) together with heat-induced delamination and fibre-matrix interfacial cracking (Fig. 7a&b). The glass fibre laminate also experienced a large reduction in tensile failure stress with increasing heat flux exposure time, and this was due to glass transition softening of the matrix that reduced the load transfer efficiently to the load-bearing fibres. However, the temperature was too low to cause weakening of the glass fibres, and therefore the tensile strength reached a steady-state value (~170 MPa) due to the fibre carrying the applied force, even though the matrix had fully softened.

These findings clearly reveal that the tensile load-bearing capacity of plant fibre composites is rapidly and severely compromised when exposed to a moderately low temperature fire.

Fibreglass composites retain significant tensile strength after the polymer matrix has fully softened, however the plant fibre materials have negligible residual strength after several minutes due to rapid softening of the fibres and internal damage (e.g. delaminations, fibre-matrix debonding).

3.3.2 High Temperature Fire Exposure (Heat Flux = 35 kW/m²)

Fire tests performed on the plant and glass fibre composites at the higher heat flux of 35 kW/m² generated higher temperatures that caused softening and decomposition of the polymer matrix and fibre reinforcement. The effect of exposure time to the 35 kW/m² flux on the fire reaction properties (mass loss, heat release rate, smoke density) of the different composites is shown in Figure 10. The mass of all the materials decreased with increasing time, although the mass loss rate was higher for the plant fibre composites due to water evaporation followed (at higher temperatures) by decomposition of the fibres, which does not occur with E-glass. The heat release rate was also higher for the natural fibre composites, and this was due to the higher

organic content of these materials together with the higher internal temperatures when compared to the glass fibre laminate. A distinguishing feature of the heat release rate curves is they did not display a distinct sharp peak, and this is because the materials did not ignite and undergo flaming combustion which typically causes the sharp peak. Instead, the plant fibre composites experienced internal smouldering combustion. Despite the higher mass loss and heat release rates, the natural fibre composites had similar smoke specific extinction area (SEA) values to the glass fibre laminate. The smoke specific extinction (SEA) area is a measure of the instantaneous amount of smoke produced per unit mass of the combusted material. The measured values were similar because most of the smoke was generated by decomposition and vapourisation of the vinyl ester matrix, which is present in the same quantity in all the composites.

The effect of increasing exposure time to the 35 kW/m^2 flux on the temperatures at the front, centre and back of the composites is shown in Figure 11. The temperature of the front surface increased rapidly and at the same rate when the composites were initially exposed to the heat flux (up to $\sim 200 \text{ s}$), after which there were significant differences between the materials. The temperatures of all the plant fibre composites rose continuously with heat flux exposure time, whereas the glass fibre laminate reached a steady-state temperature. Also, the temperatures at the centre and back surface of the plant fibre composites exceeded their front surface, which did not occur with the glass fibre laminate. During testing it was observed that the hemp, jute and flax fibres below the front surface experienced smouldering combustion. The plant fibres were hot enough to decompose and glow, however the oxygen content below the composite surface was too low for them to undergo flaming combustion. The pressure generated by the outward flow of water vapour and hot gases created by thermal decomposition of the plant fibres and polymer matrix was higher than ambient pressure at the heated surface, which restricted the flow of air into the composites needed to promote flaming combustion. Smouldering combustion is caused mostly by cellulose in the plant fibres [19]. Hemp has a higher cellulose content than both flax

and jute (Table 1), and therefore the higher smouldering temperatures account for this composite having the highest front face temperature.

The effect of increasing exposure time to the heat flux of 35 kW/m^2 on the tensile failure stresses of the composites is shown in Figure 12. As expected, plant fibre composites were vastly inferior to the glass fibre laminate. The plant fibre composites failed rapidly, even at low stress, whereas the glass fibre material withstood much higher stresses for longer heat flux exposure times.

Similar to the lower heat flux, the flax fibre composite had higher tensile strength compared to the laminates containing hemp or jute fibres (which had similar performance). The rapid weakening and failure of the plant fibre composites was due primarily to thermal softening of the polymer matrix and fibres, and not the effects of decomposition and smouldering combustion (which occur at higher temperatures). This is proven by Figure 13, which compares the effect of increasing exposure time to the 35 kW/m^2 flux on the failure stress and mass loss rate of the hemp fibre composite. (Similar trends were observed for the flax and jute composites). The tensile failure stress of the hemp composite fell rapidly (within the initial ~ 250 seconds) when exposed to the heat flux. However, thermal decomposition of the hemp fibres and polymer matrix occurred over much longer times, as defined by the increase in the mass loss rate curve. Less than a few percentage of the plant fibre composite had decomposed before it had lost virtually all its load-bearing capacity. In contrast, the glass fibre laminate had residual strength during and after complete decomposition of the polymer matrix. The mass loss rate curve in Figure 13 shows that the matrix to the fibreglass laminate had fully decomposed after ~ 2500 seconds, however the material had residual strength (approx. 50 MPa) for much longer times due to the glass reinforcement. This reveals that the glass fibres provide load-bearing properties during and even after decomposition of the matrix phase, which does not occur with plant fibre composites.

4 CONCLUSIONS

Plant fibre composites have inferior fire resistance compared to an E-glass laminate with the same fibre content when exposed to heat fluxes representative of low and moderate temperature fires. The inferior fire resistance can be attributed to water evaporation, glass transition softening and damage (delamination, fibre-matrix interfacial debonding) that occurs to plant fibre composites at moderately low temperatures (typically under 200-250°C). The tensile properties of plant fibre composites are severely weakened before any significant decomposition occurs to the matrix or fibres. However, the fire performance of plant fibre composites is not all the same, and in this study the flax laminate had higher tensile strength compared to the jute and hemp materials (which were similar). Based on this study, the increasing use of plant fibres in place of E-glass as the reinforcement to polymer composites comes at the increased risk of much higher heat release rates, which increases the fire safety hazard, and inferior fire tensile properties, which increases the risk of structural failure. This is an important reason for the need of flame retardant additives in plant fibre composites.

The experimental results and findings presented in this work are for the case when the fire is radiating heat from the flame on to the composite surface, however there is no direct contact with the flame. It is possible that direct flame impingement may affect the fire structural response of the composites by altering the thermal boundary condition of the front surface, although the main findings are expected to be similar to the condition studied here for a radiant heat flux.

ACKNOWLEDGEMENTS

This work was undertaken as part of the Composites Fire Performance project of the Cooperative Research Centre for Advanced Composite Structures Ltd (CRC-ACS), established and supported under the Australian Government's Cooperative Research Centres Program. One of the authors (T.B.) thanks the CRC-ACS for the provision of a PhD scholarship. The authors

thank Robert Ryan, Peter Tkatchyk and Nadia Zakhartchouk from RMIT University for technical assistance in the manufacture and testing of the composites, respectively.

REFERENCES

1. Carus M, Scholz L. Targets for bio-based composites and natural fibres. 8th ed. Germany: European Industrial Hemp Association, 2010.
2. Farnfield CA, Alvey PJ. Textile terms and definitions. 7th ed. Manchester: The Textile Institute, 1975.
3. Alix S, Philippe E, Bessadok A, Lebrun L, Morvan C, Marais S. Effect of chemical treatments on water sorption and mechanical properties of flax fibres. *Bioresource Technology* 2009;100(20):4742-4749.
4. Baley C, Busnel F, Grohens Y, Sire O. Influence of chemical treatments on surface properties and adhesion of flax fibre–polyester resin. *Composites Part A: Applied Science and Manufacturing* 2006;37(10):1626-1637.
5. le Duigou A, Bourmaud A, Balnois E, Davies P, Baley C. Improving the interfacial properties between flax fibres and PLLA by a water fibre treatment and drying cycle. *Industrial Crops and Products* 2012;39:31-39.
6. Stamboulis A, Baillie CA, Peijs T. Effects of environmental conditions on mechanical and physical properties of flax fibers. *Composites Part A: Applied Science and Manufacturing* 2001;32(8):1105-1115.
7. Dittenber DB, GangaRao HVS. Critical review of recent publications on use of natural composites in infrastructure. *Composites Part A: Applied Science and Manufacturing* 2012;43(8):1419-1429.
8. Pil L, Bensadoun F, Pariset J, Verpoest I. Why are designers fascinated by flax and hemp fibre composites? *Composites Part A: Applied Science and Manufacturing* 2016;83:193-205.
9. Yan L, Chouw N, Jayaraman K. Flax fibre and its composites – A review. *Composites Part B: Engineering* 2014;56:296-317.
10. Dhakal HN, Zhang Z. The use of hemp fibres as reinforcements in composites. In: Faruk O, Sain M, editors. *Biofiber Reinforcements in Composite Materials*: Woodhead Publishing; 2015. p. 86-103.
11. Gopinath A, Kumar MS, Elayaperumal A. Experimental investigations on mechanical properties of jute fiber reinforced composites with polyester and epoxy resin matrices. *Procedia Engineering* 2014;97:2052-2063.
12. Ingrao C, Lo Giudice A, Bacenetti J, Tricase C, Dotelli G, Fiala M, Siracusa V, Mbohwa C. Energy and environmental assessment of industrial hemp for building applications: A review. *Renewable and Sustainable Energy Reviews* 2015;51:29-42.
13. Khan JA, Khan MA. The use of jute fibers as reinforcements in composites. In: Faruk O, Sain M, editors. *Biofiber reinforcements in composite materials*: Woodhead Publishing; 2015. p. 3-34.
14. Misnon MI, Islam MM, Epaarachchi JA, Lau KT. Analyses of woven hemp fabric characteristics for composite reinforcement. *Materials & Design* 2015;66, Part A:82-92.
15. Mohanty AK, Misra M, Drzal LT. Surface modifications of natural fibers and performance of the resulting biocomposites: An overview. *Composite Interfaces* 2001;8(5):313-343.
16. Moriana R, Vilaplana F, Karlsson S, Ribes A. Correlation of chemical, structural and thermal properties of natural fibres for their sustainable exploitation. *Carbohydrate Polymers* 2014;112:422-431.

17. Müssig J, Haag K. The use of flax fibres as reinforcements in composites. In: Faruk O, Sain M, editors. *Biofiber Reinforcements in Composite Materials*: Woodhead Publishing; 2015. p. 35-85.
18. Wambua P, Ivens J, Verpoest I. Natural fibres: can they replace glass in fibre reinforced plastics? *Composites Science and Technology* 2003;63(9):1259-1264.
19. Chapple S, Anandjiwala R. Flammability of natural fiber-reinforced composites and strategies for fire retardancy: A review. *Journal of Thermoplastic Composite Materials* 2010;23(6):871-893.
20. Kandare E, Luangtriratana P, Kandola BK. Fire reaction properties of flax/epoxy laminates and their balsa-core sandwich composites with or without fire protection. *Composites Part B: Engineering* 2014;56:602-610.
21. Manfredi LB, Rodríguez ES, Wladyka-Przybylak M, Vázquez A. Thermal degradation and fire resistance of unsaturated polyester, modified acrylic resins and their composites with natural fibres. *Polymer Degradation and Stability* 2006;91(2):255-261.
22. Mngomezulu ME, John MJ, Jacobs V, Luyt AS. Review on flammability of biofibres and biocomposites. *Carbohydrate Polymers* 2014;111:149-182.
23. Chai S, Bickerton S, Bhattacharyya D, Das R. Influence of natural fibre reinforcements on the flammability of bio-derived composite materials, *Comp B.*, 2012;43:2867-2874.
24. Naughton A, Fan M, Bregulla J. Fire resistance characterisation of hemp fibre reinforced polyester composites for use in the construction industry. *Composites Part B: Engineering* 2014;60:546-554.
25. Chapple S, Anandjiwala R. Flammability of natural fiber-reinforced composites and strategies for fire retardancy: A review. *Journal of Thermoplastic Composite Materials* 2010.
26. Subasinghe A, Bhattacharyya D. Performance of different intumescent ammonium polyphosphate flame retardants in PP/kenaf fibre composites; *Comp A*, 2014;65:91-99.
27. Kim, Subasinghe A, Bhattacharyya D. Natural fibres: Their composites and flammability characterizations; in *Multi-functionality of Polymer Composites: Challenges and New Solutions*, ed. K Friedrich and U Brewer, Elsevier, Waltham, MA, USA, 2015, pp. 102-143.
28. Gourier C, Le Duigou A, Bourmaud A, Baley C. Mechanical analysis of elementary flax fibre tensile properties after different thermal cycles. *Composites Part A: Applied Science and Manufacturing* 2014;64:159-166.
29. Feih S, Boiocchi E, Mathys G, Mathys Z, Gibson AG, Mouritz AP. Mechanical properties of thermally-treated and recycled glass fibres. *Composites Part B: Engineering* 2011;42(3):350-358.
30. Feih S, Mouritz AP. Tensile properties of carbon fibres and carbon fibre-polymer composites in fire. *Composites Part A: Applied Science and Manufacturing* 2012;43(5):765-772.
31. ASTM D3171- 11 Standard Test Method for Constituent Content of Composite Materials 2011.
32. ASTM D638 - 10 Standard Test Method for Tensile Properties of Plastics. PA, United States 2010.
33. Bausano JV, Lesko JJ, Case SW. Composite lifetime during combined compressive loading and one-sided simulated fire exposure: characterization and prediction, *Comp.*, 2006;37A:1092-1100.
34. Boyd SE, Case SW, Lesko JJ. Compression creep rupture behavior of a glass/vinyl ester composite subject to isothermal and one-sided heat flux conditions. *Comp.*, 2007;38:1462-1472.
35. Gibson AG, Wu Y-S, Evans JT, Mouritz AP. Laminate theory analysis of composites under load in fire, *J. Comp. Mat*, 2006;40:639-658.

36. Easby RC, Feih S, Konstantis C, La-Delfa G, Urso Miano V, Elmughrabi A, Mouritz AP, Gibson AG, A failure model for phenolic and polyester pultrusions under load in fire, *Plas. Rubb. & Comp.*, 2007;36:379-388.
37. Keller T, Tracy C, Hugi E. Fire endurance of loaded and liquid-cooled GFRP slabs for construction. *Comp A*. 2006;37:1055-1067.
38. Correia JR, Branco FA, Ferreira JG, Bai Y, Keller T. Fire protection systems for building floors made of pultruded GFRP profiles Part 1: Experimental investigations. *Comp B*, 2010;41:617-629.
39. Liu L, Kardomateas GA, Birman V, Holmes JW, Simites JG. Thermal buckling of a heat exposed, axially restrained composite column, *Comp A*, 2006;37:972-980.
40. Griffis CA, Nemes JA, Stonesifer FR, Chang CI. Degradation in strength of laminated composites subjected to intense heating and mechanical loading. *J. Comp. Mat.*, 1986;20: 216-235.
41. Pering GA, Farrell PV, Springer GS. Degradation of tensile and shear properties of composites exposed to fire or high temperature. *J. Comp. Mats*, 1989;14:54-66.
42. Asaro RJ, Lattimer B, Ramroth W. Structural response of FRP composites during fire, *Comp. Struct.*, 2009; 87, 382-393.
43. Gibson AG, Wright PNH, Wu Y-S, Mouritz AP, Mathys Z, Gardiner CP. The integrity of polymer composites during and after fire, *J Comp. Mat.*, 2004;38:1283-1308.
44. Feih S, Mathys Z, Gibson AG, Mouritz AP, Modelling the tension and compression strengths of polymer laminates in fire, *Comp. Sci. & Tech.*, 2007;67:551-564.
45. Feih S, Mathys Z, Gibson AG, Mouritz AP. Modelling the compression strength of polymer laminates in fire, *Comp.*, 2007;38A:2354-2365.
46. ISO 5660 - 1: 2015 Reaction-to-fire tests - Heat release, smoke production and mass loss rate - Part 1: Heat release rate (cone calorimeter method) and smoke production rate (dynamic measurement) 2015.
47. Kozłowski R, Władyka-Przybylak M. Flammability and fire resistance of composites reinforced by natural fibers. *Polymers for Advanced Technologies* 2008;19(6):446-453.
48. Van de Velde K, Baetens E. Thermal and mechanical properties of flax fibres as potential composite reinforcement. *Macromolecular Materials and Engineering* 2001;286(6):342-349.
49. Feih S, Mouritz AP, Case SW. Determining the mechanism controlling glass fibre strength loss during thermal recycling of waste composites. *Composites Part A: Applied Science and Manufacturing* 2015;76:255-261.
50. Baley C, Le Duigou A, Bourmaud A, Davies P. Influence of drying on the mechanical behaviour of flax fibres and their unidirectional composites. *Composites Part A: Applied Science and Manufacturing* 2012;43(8):1226-1233.
51. Baley C, Morvan C, Grohens Y. Influence of the absorbed water on the tensile strength of flax fibers. *Macromolecular Symposia* 2005;222(1):195-202.
52. Gassan J, Bledzki AK. Thermal degradation of flax and jute fibers. *Journal of Applied Polymer Science* 2001;82(6):1417-1422.
53. Tajvidi M, Takemura A. Thermal degradation of natural fiber-reinforced polypropylene composites. *Journal of Thermoplastic Composite Materials* 2010;23(3):281-298.
54. Baley C. Analysis of the flax fibres tensile behaviour and analysis of the tensile stiffness increase. *Composites Part A: Applied Science and Manufacturing* 2002;33(7):939-948.
55. Girault R, Bert F, Rihouey C, Jauneau A, Morvan C, Jarvis M. Galactans and cellulose in flax fibres: putative contributions to the tensile strength. *International Journal of Biological Macromolecules* 1997;21(1-2):179-188.
56. Goodrich T, Nawaz N, Feih S, Lattimer BY, Mouritz AP. High-temperature mechanical properties and thermal recovery of balsa wood. *Journal of Wood Science* 2010;56(6):437-443.
57. Ramiah MV. Thermogravimetric and differential thermal analysis of cellulose, hemicellulose, and lignin. *Journal of Applied Polymer Science* 1970;14:1323-1337.

58. Szczesniak L, Rachocki A, Tritt-Goc J. Glass transition temperature and thermal decomposition of cellulose powder. *Cellulose* 2008;15:445-451.
59. Beukers A, Ed van Hinte. *Lightness: The Inevitable Renaissance of Minimum Energy Structures*. 4 ed. Rotterdam: 010 Publishers, 2005.
60. Bos HL, Van Den Oever MJA, Peters OCJJ. Tensile and compressive properties of flax fibres for natural fibre reinforced composites. *Journal of Materials Science* 2002;37(8):1683-1692.
61. Sutton A, Black D, Walker P. *Natural Fibre Insulation - An introduction to low-impact building materials*. 2011 [cited 2016 01 April]; Available from: http://www.bre.co.uk/filelibrary/pdf/projects/low_impact_materials/IP18_11.pdf.
62. Feih S, Mouritz AP, Mathys Z, Gibson AG. Tensile strength modeling of glass fiber-polymer composites in fire. *Journal of Composite Materials* 2007;41(19):2387-2410.

Figure 1: Schematic of test to measure high temperature tensile properties of fibre tows.

Figure 2. Schematic of the fire structural test (side-view).

(a)

Figure 3: Effect of temperature on the (a) tensile failure load and (b) tensile stiffness of the tows. The load and stiffness values have been normalised to the tex value of the tow. The curves are lines-of-best fit through the tensile property data.

(a)

(b)

Figure 4: Effect of temperature on the (a) tensile strength and (b) tensile modulus of the plant and glass fibre composites. The curves are lines-of-best fit through the tensile property data.

Figure 5. Effect of heat flux exposure time on the heat release rate of the composites at the flux of 10kW/m².

(a)

(b)

Figure 6. Temperature-time curves for the (a) front and (b) back surfaces of the composites exposed to the heat flux of 10 kW/m^2 .

(a)

(b)

(c)

Figure 7. Scanning electron microscope images of (a) delamination cracking and (b) fibre-matrix interfacial debonding in the hemp fibre composite. (c) Delamination in the glass fibre composite. Heat flux of 10 kW/m^2 .

Figure 8. Mass loss curves for the composites exposed to the heat flux of 10 kW/m^2 .

Figure 9. Effect of heat flux exposure time on the tensile failure stress of the composites at the heat flux of 10 kW/m^2 . The curves are lines-of-best fit.

(a)

(b)

(c)

Figure 10. Effect of heat flux exposure time on the (a) mass loss, (b) heat release rate and (b) smoke density (SEA) of the composites at the heat flux of 35 kW/m².

(a)

(b)

(c)

Figure 11. Temperature-time curves for the (a) front surface (b) mid-thickness and (c) back surface of the composites exposed to the heat flux of 35 kW/m^2 .

Figure 12. Effect of heat flux exposure time on the tensile failure stress of the composites. Heat flux of 35 kW/m^2 . The curves are lines-of-best fit.

(a)

(b)

Figure 13. Effect of heat flux exposure time on the failure time and mass loss rate of the (a) hemp fibre composite and (b) glass fibre composite. Heat flux = 35 kW/m².

Table 1: Percentage fractions of the main organic constituents in flax, jute and hemp fibres [7].

	Hemicellulose (wt%)	Cellulose (wt%)	Lignin (wt%)	Pectin (wt%)	Waxes (wt%)
Flax	18.6-20.6	62-72	2-5	2.3	1.5-1.7
Hemp	15-22	68-74	3.7-10	0.9	0.8
Jute	13.6-20.4	59-71	11.8-13	0.2-0.4	0.5