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## Experimental tools applied to the ignition study of spruce wood under cone calorimeter

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### ABSTRACT

Wood-based materials, which are more and more used in building construction, are often characterized by normative tests using the cone calorimeter. The ignition time and temperature, the mass loss rate or heat release rate are the most current measurements performed. The sample ignition can vary according to a large number of variables (humidity, density, scale sample, experimental conditions). The auto ignition phenomenon has been studied so far but opinions concerning the involved physical processes remain controversial. The purpose of the present work is to improve the study of thermal degradation of wood under cone calorimeter thanks to a dedicated experimental set up. This set up includes mainly, an infrared camera, enabling an accurate surface temperature measurement and a fast visible camera (3000 fps), used to highlight the “auto” ignition phenomenon. Surface temperature and mass loss rate evolution are presented for different heat flux expositions. The fast visible camera pictures showed an ignition piloted by the coil. The ignition occurs only above  $55 \text{ kW.m}^{-2}$ . When this external heat flux was sufficiently low, it was possible to observe the transition between pyrolysis solely and smouldering combustion for a sample surface temperature around  $400^\circ \text{C}$ .

### KEYWORDS

*Cone calorimeter; ignition; surface temperature; flame spread.*



## INTRODUCTION

With the ecological transition and the modernisation of urban planning, uses of wood-based material as construction structures or facades becomes increasingly popular. Among available solutions, wood appears to be an interesting material because of its mechanical properties. For fire safety concerns in construction domain, the study of fire behaviour and flame propagation in wood structures is important due to its flammability. Nevertheless, some previous studies have shown that a good choice of wood exhibits encouraging resistance to fire [1, 2], especially a slow in-depth propagation and an ability to self-extinguishment.

The fire behaviour of wood has been widely studied for several years, mainly the ignition criteria [3]. When the sample surface is exposed to an external heat flux or an external flame, pyrolysis occurs within the sample releasing pyrolysis gases. Pyrolysis process depends on several parameters such as moisture, density, heat flux, experimental conditions and so on, and it is consequently very difficult to clearly identify and characterise “the ignition conditions” [4, 5, 6], even if it is well known that ignition occurs when the pyrolysis products, at a sufficient concentration, reach their ignition point. The most difficult part remains to experimentally determine this ignition point. Moreover, after ignition, the additional heat provided by flames on the surface helps to sustain or even to hasten the pyrolysis process. Before their usage, building materials are subjected to different fire normative tests. The cone calorimeter test is one of them. It is a standard test (ASTM and ISO) useful for measuring the Heat Release Rate (HRR) of materials as a function of calibrated incident fluxes. Many fire models need to link this HRR to the net heat flux into the surface in order to best describe the degradation process.

This net heat flux can be calculated provided the measure of surface and in-depth temperature during the test [7]. This test, at a small scale, can be done in two orientations (vertically or horizontally) even if the horizontally orientation was often preferred for practical reasons. However, studies showed that vertical orientation seems more critical than the horizontal one [888, 9]. As stated by Babrauskas [3], physical data such as temperature and time to ignition seemed to give a wide variety of results. It is getting worse if we also consider the time to incandescence [10]. This time could be confused with the time of ignition when the heat flux is high. The surface temperature measured at this time is often considered to be the ignition temperature.

The aim of the present work is to present some experimental tools applied to the study of the auto-ignition of wood under exposure of an external heat flux provided by a vertically oriented cone calorimeter. The ignition of the wood will be highlighted using a fast visible camera. A precision scale is used in order to record the mass loss during the test. In addition, an infrared camera is used to observe the radiation from the surface of wood samples during thermal degradation experiments. Infrared images are post-processed involving a subtraction method between successive images, to withdraw the high incident flux from the cone calorimeter partly reflected by the sample. Then, an identification step was implemented linking the rise in intensity between two time steps with the increase in sample surface temperature.

## MATERIALS AND METHODS

### Samples and cone calorimeter test

The selected wood material was spruce. The sample sizes were  $100 \times 100$  mm with a 50 mm thickness. The sample average density was about  $480 \text{ kg m}^{-3}$ , given with an average moisture content around 9 %. As specified in the standard ISO 5660-1, the samples were wrapped with two layers of aluminium foil, apart from the top side exposed to the radiative flux. The distance between the sample and the heater was 25 mm. The samples were exposed vertically to an external constant heat flux between 15 and  $75 \text{ kW.m}^{-2}$ . This radiative heat flux emitted by the cone was controlled before each test thanks to a Schmidt-Boelter fluxmeter (Medtherm). Cone emission could be assumed to be close to those of a blackbody [11]. The surface temperature was measured as explained below. A sample was considered ignited when a visible flame was observed.

### Temperature measurement

Surface or ignition temperatures are usually measured using thermocouples. However, these measurements are punctual and could be false due to the heat sink along the thermocouple wire or to bad contact between the thermocouple and the sample. This contact seems all the more difficult for the wood that is degrading, produces char and cracks [12]. In this work, surface temperatures will be measured using a multispectral infrared camera (Orion SC7000 by FLIR). This non-intrusive measurement allows studying the temperature field evolution on a large surface. The contact problem is also eliminated. During infrared measurements, the flame between the infrared camera and the sample surface may lead to a mismeasurement of the temperature.

Some studies have shown that the emission by flames is observed to be predominant in specific wavelengths where major combustion gases ( $\text{CO}_2$  and  $\text{H}_2\text{O}$ ) can emit [13]. Outside these bands, only soot can emit, and since the optical thickness for the flames that are involved in the cone calorimeter experiments is very small, the flame can be considered as almost transparent. The camera is equipped with a filter wheel to select wavelengths. In the present paper, the selected wavelength was  $3.9 \mu\text{m}$  ( $2564 \text{ cm}^{-1}$ ), which is outside the emission bands of combustion gases. This type of measurement was already successfully performed by our team on other materials [14]. Once the camera signal is acquired, it is converted to a spectral intensity, thanks to a preliminary calibration of the camera involving a blackbody, and the temperature is calculated using the inversion of the Planck's law. The emissivity for wood was studied in [15]. It is shown in this paper that the emissivity of wood at  $2564 \text{ cm}^{-1}$  is close to 0.9 and that it doesn't vary more than  $\pm 5\%$  around this value when the wood deteriorates under a heat flux. For Chaos et al, a misestimate of the emissivity in 0.8-1 range can lead to an error of 8% in the surface temperature [16]. Fig 1 shows the temperature field obtained for two different tests ( $20$  and  $55 \text{ kW m}^{-2}$ ) at the same time. The white circle is the study area where the mean surface temperature was calculated.

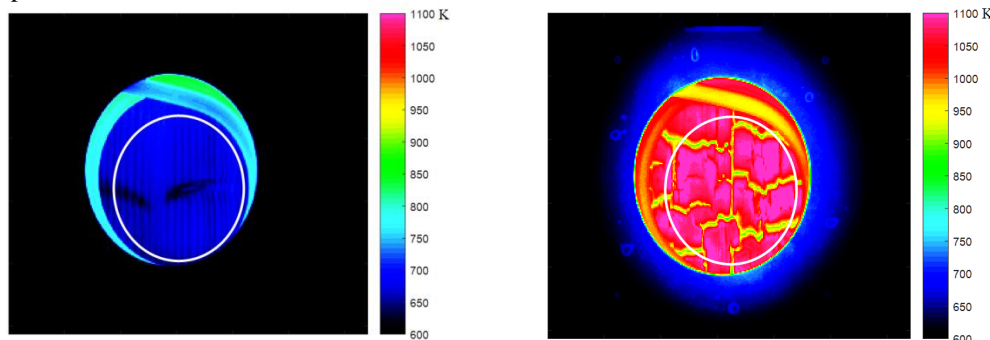


Fig. 1. Temperature field in Kelvin and studied area (white circle) at 120 s for two different external heat fluxes ( $20 \text{ kW.m}^{-2}$  (left) and  $55 \text{ kW.m}^{-2}$  (right)).

The time evolution of the surface temperature obtained from the IR camera and a K-type thermocouple (1 mm diameter) are plotted in Fig 2. In a general way, the surface temperature increases more or less quickly before reaching a plateau. Until 1500 s, the temperature given by the thermocouple and the infrared camera are in pretty good agreement, even if the thermocouple underestimates the surface temperature. The discrepancy between the two measurements increases for large time. Given that the sample surface burns, the contact between the thermocouple and the surface can no longer be ensured, resulting in a decrease of the temperature. In addition, the thermocouple signal becomes more and more “noisy” since it is subjected to the temperature variation of surrounding, caused by the flame notably. Although widely used, we show here that surface temperature measurement by thermocouple can lead to great uncertainty. For the sake of accuracy, surface temperature measurement by IR camera should be preferred. This, of course, means that radiative properties have to be well known, especially the sample emissivity, which may vary during the degradation process. A parametric study of the sample emissivity was also done. Three values of emissivity (0.88, 0.90 and 0.95) were chosen in the typical range of variation of wood emissivity during its thermal degradation, as reported by Boulet et al. [15]. For the two tests at  $20$  and  $55 \text{ kW.m}^{-2}$ , the temperature range due to the emissivity uncertainty holds within  $20^\circ\text{C}$  in the worst case, which is very reasonable. For future work we plan to use a multispectral thermography in order to estimate both temperature and emissivity.

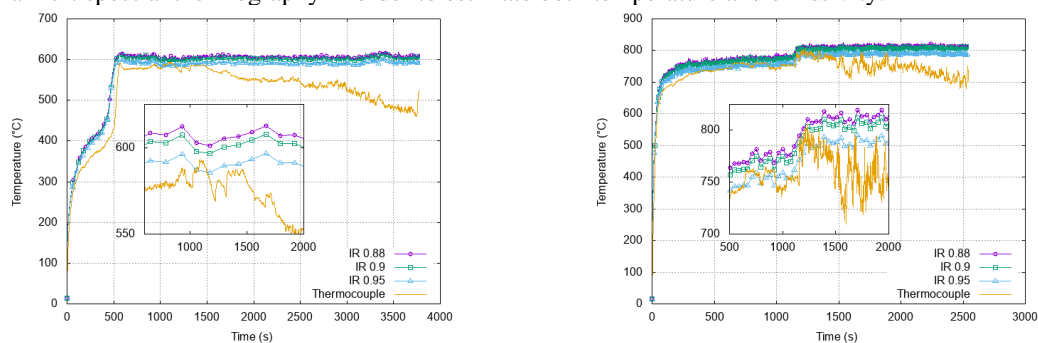


Fig. 2. Time evolution of the surface temperature for two different external heat fluxes ( $20 \text{ kW.m}^{-2}$  (left) and  $55 \text{ kW.m}^{-2}$  (right)) calculated with the infrared camera and measured with a thermocouple located at the center of the surface .

## RESULTS AND DISCUSSION

Samples were exposed to five external heat fluxes (15, 20, 45, 55, 75 kW.m<sup>-2</sup>). Flame ignition was only present for the tests at 55 and 75 kW.m<sup>-2</sup>.

### Samples with smouldering combustion (no flame)

The cone coil temperature was set to 770 °C for obtaining a desired radiative heat flux of 45 kW.m<sup>-2</sup>, to 570 °C for 20 kW.m<sup>-2</sup>, and to 520 °C for 15 kW.m<sup>-2</sup>. For tests with external heat fluxes of 15 and 20 kW.m<sup>-2</sup>, the total exposition time was respectively 8000 s and 3600 s. During these tests, no flame appeared but smouldering combustion phenomenon was observed as discussed below. Figure 3 shows the Mass Loss Rate (MLR) and surface temperature evolution provided by IR camera acquisitions. Since the MLR and the surface temperature remain constant after 2000 s, the curves are plotted only from 0 to 3600 s.

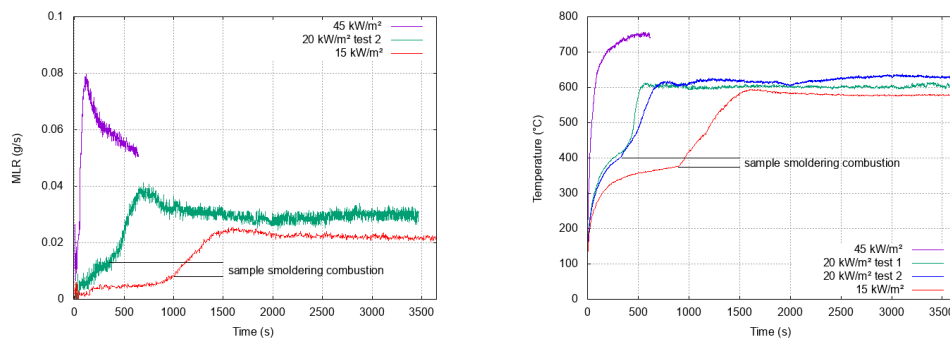


Fig. 3. MLR and surface temperature for tests without sample ignition (15, 20 and 45 kW.m<sup>-2</sup>).

In a general way the MLR first increases until a maximum. Then the MLR decreases, as the char plays the role of an insulator layer that reduce heat transfer within the sample, before reaching a plateau. For the test at 15 and 20 kW.m<sup>-2</sup>, an acceleration of the MLR is observed at 900 s and 345 s respectively. This acceleration is also observed for the temperature for which the transition occurs around 370 and 410 °C respectively. This phenomenon corresponds to the beginning of the smouldering combustion of the char that leads to an additional amount of energy release. The lower the external heat flux is, the more the transition between the pyrolysis without char combustion and pyrolysis accompanied by smouldering combustion can be remarked. For the test at 45 kW.m<sup>-2</sup> this transition is hardly visible because the MLR and temperature increase is too fast.

### Samples with presence of flame

Tests were also carried out with higher radiative heat fluxes for which the ignition occurs. Fig 4 shows the MLR (left) and the surface temperature (right) evolutions for tests under 55 and 75 kW.m<sup>-2</sup>. The cone coil temperature was set to 825 °C and 920 °C respectively.

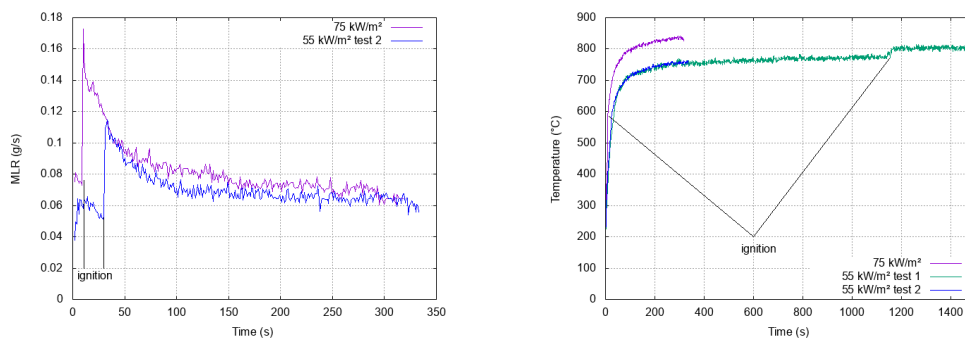


Fig. 4. MLR and surface temperature for tests with sample ignition (55 and 75 kW.m<sup>-2</sup>).

Similar overall trends as previous tests without ignition are observed. The main difference is a drastic increase in the MLR and the surface temperature when the ignition occurs. This is clearly pointed out in the MLR curves of the figure 4. This increase is all the more pronounced that the flux is high. For the test 75 kW.m<sup>-2</sup>, the MLR is doubled from 0.08 to 0.17 g s<sup>-1</sup>. The ignition occurs at 11 s for the test at 75 kW.m<sup>-2</sup> and at 30 s for the test at 55 kW.m<sup>-2</sup>. The corresponding surface temperature were estimated to 609 °C for 75 kW m<sup>-2</sup> and to



776 °C and 618 °C for the two tests at 55 kW m<sup>-2</sup>. Besides some heterogeneities of each sample, the experiment conditions, in particular the flame origin can affect results. After the ignition, the MLR decreases due to the protecting role plays by the char, as discussed previously.

### Ignition of the sample

As explained above, the ignition time corresponds to the moment when a flame appears on the sample surface. At this time, the surface temperature is often called ignition temperature. However, it is difficult to explain the conditions which lead to the ignition. We planned to find out if ignition corresponds to a real auto ignition as it is often claimed or if it is rather piloted by the fact that pyrolysis gases encounter the hot cone coil. To answer this question, a fast camera was placed to observe the sample ignition. The camera was set with 3000 frames per second, which allowed to record 1.3 seconds. The recorded pictures during ignition are presented in figure 5 and 6 for two tests, performed respectively at 55 and 75 kW.m<sup>-2</sup>. As suspected, the gases ignition is well caused by the cone coil and not by the sample itself. Then, the corresponding surface temperature can no longer be really considered as the ignition temperature.

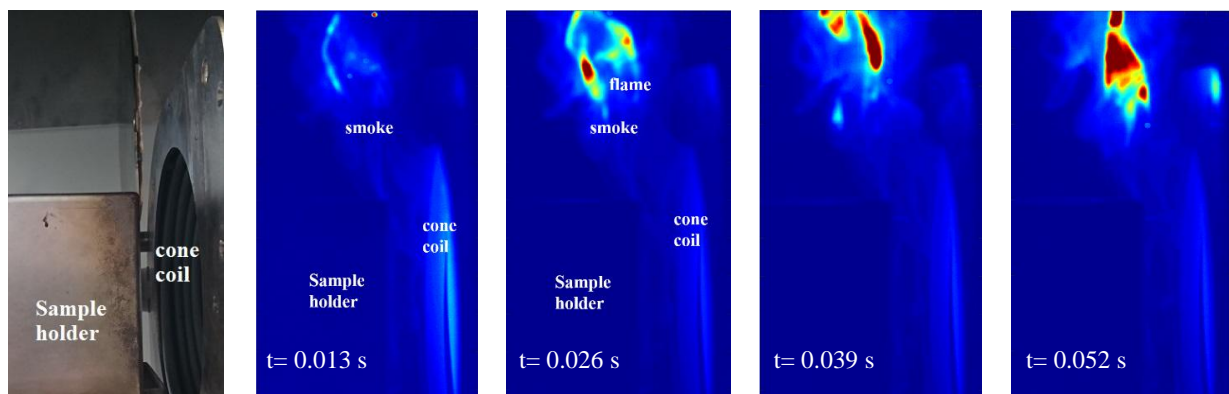


Fig. 5. Ignition at 55 kW.m<sup>-2</sup>.

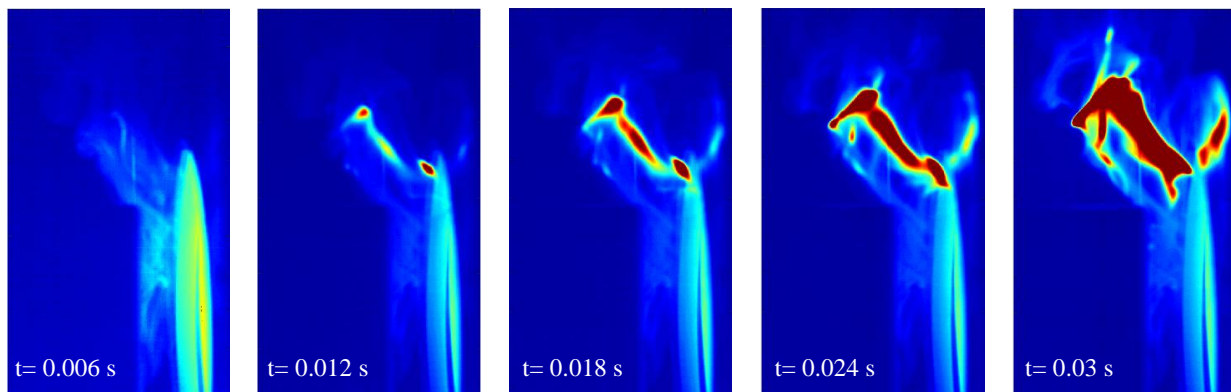


Fig. 6. Ignition at 75 kW.m<sup>-2</sup>.

### CONCLUSION

A dedicated experimental set up was used in order to enhance the thermal degradation study of spruce wood under cone calorimeter. A fast visible camera was used in order to best describe the ignition phenomenon. A significant progress was made by stating that the ignition occurs when the pyrolysis gases impacting the hot cone coil. The surface temperature was also measured with an infrared camera. This temperature is determined using a filter to overcome the presence of the flame. The calculation was done by inverting Planck's law, fixing the emissivity. In future tests, more filters at different wavelengths will be used in order to improve the accuracy of the surface temperature calculation. This step will also make it possible to take into account the emissivity evolution over time.

Thermocouples will be then placed inside the sample to deduce the char presence and to calculate the conductive flux. The char layer prediction is crucial for the design of wood structure. In first tests, pyrolysis

was immediately stopped with liquid nitrogen in order to get an idea of the proportion of char layer. Then, the proportion of wood consuming by smouldering could be studied.

Some studies had shown that the sample moisture had a significant influence on the temperature, time to ignition, and pyrolysis. In future tests the wood moisture will be increased up to 30 %. Finally, the set up will be further improved in order to study the extinction phenomenon too.

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