



## Surface coated ZnO powder as flame retardant for wood: A short communication



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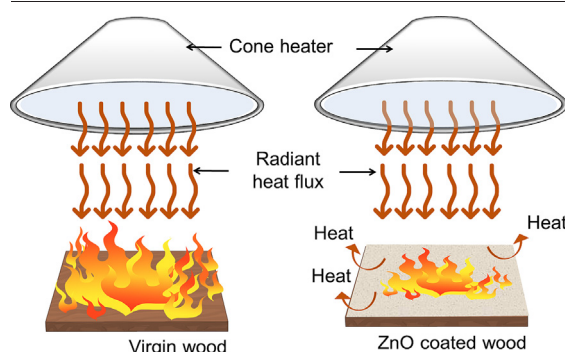
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### HIGHLIGHTS

- Assessment of the ability of ZnO coated wood to withstand radiative heat.
- Various coating thicknesses and heat fluxes from cone calorimeter were tested.
- ZnO can delay the time to ignition and it is proportional to the coating thickness.

### GRAPHICAL ABSTRACT



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### ABSTRACT

In the present study, the ability of a coating of zinc oxide (ZnO) powder to improve the fire-safety of wood exposed to radiative heat flux was examined, focusing on the ignition time of the wood. To test ZnO's efficiency on the wood substrate, two different amounts of ZnO (0.5 and 1 g ZnO per dm<sup>2</sup>) were applied to the wood surface and exposed to radiative heat from a cone calorimeter wherein a pristine piece of wood with no ZnO treatment was taken as control. The experiments were conducted at three different irradiation levels i.e., 20, 35, and 50 kWm<sup>-2</sup>. The results showed that applying ZnO on the surface of the wood significantly increased the ignition time (TTI). For the three different heat fluxes, using 0.5 g ZnO per dm<sup>2</sup> coating on the wood surface increased the TTI by 26–33 %. Furthermore, the application of 1 g of ZnO per dm<sup>2</sup> generated a TTI increment of 37–40 %. All three irradiation levels showed similar trends in TTI. The micrographs taken before and after combustion showed no significant disparity in the morphology of ZnO. The agglomerated ZnO particles on the wood surface remained intact after combustion. This study demonstrates a facile method of using ZnO to delay the ignition of wood. This could potentially impart fire-safety to wooden structures/façades in wildland-urban interfaces and elsewhere by reducing flame spread.

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## 1. Introduction

The drive for sustainable development has tremendously increased the popularity of wood as a building material. The structure of wood is highly anisotropic and complex. Wood is primarily composed of three distinct biopolymers, namely, cellulose, hemicellulose, and lignin, and it also contains small quantities of extractives (Spiridon, 2020; Burgert et al., 2016). The amount, composition, and type of these constituents can vary depending on the species of the wood and its location. Wood has several advantages over its synthetic counterparts (e.g. cementitious materials) such as providing natural insulation, requiring low energy for processing and biodegradability. Although wood is a commonly used material, its susceptibility to combustion makes it unsuitable for use in environments that have a high risk of fire (Köklükaya et al., 2015).

The combustion cycle of any substance is a process that occurs in a closed loop. When a material is exposed to a flame, it starts breaking down, i.e., decomposing, and gives off flammable gases like hydrocarbons and hydrogen. This reaction also generates heat and smoke in the condensed phase. The resultant combustible radicals combine easily with oxygen in the gas phase, which is an exothermic process that causes heat to be produced (Wilkie and Morgan, 2009; Camino et al., 1991; Sykam et al., 2021a). Parts of the heat that is generated travel farther to the substrate that is burning, which in turn sustains or intensifies the combustion. Radiation is one of the primary ways in which heat is transferred away from it. In fact, about one-third of the energy released during combustion is emitted as radiation (Försth and Roos, 2011). Hence, fire retardants (FRs), which are applied to bestow fire-safety to materials and structures, need to be capable of combating the radiative heat flux.

When ignited, wood, in particular, produces flammable radicals due to the high concentration of hydrocarbons it contains. To increase the fire safety of wood, the most effective method is to apply a coating that contains flame retardants (FRs) onto the wood surface. FRs have been categorised according to their active elemental composition as halogenated (Das et al., 2020), phosphorus-based (Sykam et al., 2021b), nitrogen-based (Alongi et al., 2013), boron-based (Shen, 2014), metal hydroxide-based (Guo et al., 2017), functionalised bio-char (Das et al., 2023) and so on. Although halogenated flame retardants (FRs) are highly effective at reducing the risk of fire, they have come under scrutiny from regulators due to their carcinogenic properties and tendency to accumulate in living organisms over time. Recently, metal oxides have become increasingly popular as non-halogenated flame retardants (FRs) due to their ability to be applied across a wide range of different materials and substrates. (Morgan, 2009; Jin and Chung, 2020; Li et al., 2018; Wu and Yang, 2011; Lin et al., 2011). ZnO could be a pertinent choice as FR owing to its ability to block radiation, and possessing thermal stability at high temperatures and due to its non-combustibility (Moezzi et al., 2012). In particular, the specific heat capacity of ZnO is also comparatively high at  $40 \text{ JK}^{-1} \text{ mol}^{-1}$  (Moezzi et al., 2012) and it also can reflect infrared radiation. These collective properties of ZnO contribute to reducing heat transfer and protecting a substrate from radiative heat (Wang et al., 2018). Researchers have looked into the use of ZnO nanoparticles on wood, in order to provide protection from UV radiation for outdoor wooden structures. The results of these studies have been positive, demonstrating the effectiveness of this approach (Grüneberger et al., 2015). ZnO has properties that make it a potentially useful material for improving the fire resistance of wood, particularly when it comes to reducing heat transfer through radiation (Sun et al., 2012a; Sun et al., 2012b). This is especially important when it comes to protecting wooden facades located in areas where wildlands meet urban areas (i.e., wildland-urban interfaces or WUI), as these structures are at higher risk of experiencing combustion from wildfires. Additionally, the application of ZnO to bestow fire-safety in wooden structures may be beneficial for countering global climate change. If a wildfire or forest fire includes nearby buildings, the overall fire intensity (i.e., the amount of energy released during the process of burning) will increase, which is directly linked to climate warming (Flannigan et al., 2000). Rendering wooden structures at WUI fire-safe could potentially also lower the total smoke production

from the wildfire, which has adverse effects on the atmosphere, environment, and humans (McKenzie et al., 2014). Finally and more importantly, creating fire-safety in wooden structures at WUI can lower the speed of flame spread and thus, consequently ameliorate the hazards linked to humans and eliminate/lower property losses (Masoudvaziri et al., 2023; Gjedrem and Metallinou, 2023). While studies have been conducted regarding the efficacy of ZnO coating on wood to impart fire-safety, the application method of depositing ZnO on the wooden surface was cumbersome (Sun et al., 2012a; Sun et al., 2012b). Hence, a facile method for applying ZnO on wooden substrates is needed followed by the determination of its efficiency against radiative heat. In the present study, the flame retardancy of ZnO by means of the radiative heat resistance capability was studied on the wood substrate. For this, ZnO was coated onto the wood substrate in varying quantities viz 0.5 and 1 g ZnO dm<sup>-2</sup>. The FR efficiency of these coatings was studied by using a cone calorimeter at various heat fluxes such as 20, 35 and 50 kWm<sup>-2</sup>. The FR parameters such as time to ignition (TTI), peak heat release rate (PHRR), total heat release (THR) smoke production rate (SPR) and total smoke production (TSP) were obtained. The morphological changes of ZnO coating before and after the cone calorimeter test were also studied by SEM analysis.

## 2. Materials and methods

### 2.1. Materials

ZnO powder (manufacturer specified size: 0.18–0.36 μm) was purchased from Rock Tumble (Suffolk, IP18 6SZ, UK) and was used as received. Spruce wood, from Norrbotten region of Sweden, was cut into square shapes for coating with ZnO and subsequent fire testing. Ethanol (CAS number: 64–17-5), for one of the coating techniques, was obtained from Merck, Sweden.

### 2.2. ZnO coating on wood

The spruce wood pieces, each measuring 100 × 100 × 10 mm<sup>3</sup>, were carefully weighed and put into an oven set to 110 °C for at least 24 h. This was done to eliminate any discrepancies in humidity that could potentially impact the results of the test. Once the wood had been dried, its weight was measured again, and the moisture content was calculated. At this point, ZnO was weighed and applied to the surface of the wood. Two different coating methods were tested, namely, ZnO and ethanol mixture, and pristine ZnO powder. The ZnO with ethanol mixture was found to be not adequate owing to the formation of uneven ZnO deposition over the wood substrate. The best outcome was achieved using pure ZnO powder, which could be easily applied to the wood surface by hand. Once the ZnO had been applied, in varying amounts ranging from approximately 0.5 to 1 g of ZnO dm<sup>-2</sup> of wood, the bottom and sides of the specimens were wrapped in aluminium foil. The coated side of the samples was then exposed to the cone heater during the cone calorimeter test.

### 2.3. Characterisation techniques

Moisture in the wood delays the TTI and can also lead to an increase in the amount of heat or energy needed to ignite the wood (Bartlett et al., 2019). Hence, the wooden pieces were dried at 110 °C for 24 h and the moisture content (μ) was measured as per Eq. (1). The moisture content of the wood specimen was about 6–8 %.

$$\mu = \frac{(\text{weight before} - \text{weight after})}{\text{weight after}} \times 100 = \text{moisture content in\%} \quad (1)$$

To conduct the cone calorimeter test, wooden specimens (measuring 100 × 100 × 10 mm<sup>3</sup>) were prepared, some of which were coated with ZnO (in amounts of 0.5 and 1 g.dm<sup>-2</sup>) and some left uncoated. The tests were carried out in accordance with ISO 5660 standards, using a Netzsch TCC 918 cone calorimeter. During testing, the specimens were subjected to

three different levels of heat flux: 20 kWm<sup>-2</sup>, 35 kWm<sup>-2</sup>, and 50 kWm<sup>-2</sup>. The sample tests were replicated three times for each formulation.

The surface morphology of the wood specimens (virgin and ZnO coated) before and after the cone calorimetric test was observed by scanning electron microscopy (SEM) analysis on JCM-6000 with a voltage of 15 kV under a high vacuum.

### 2.4. Analysis of Variance (ANOVA)

A single factor ANOVA ( $\alpha = 0.05$ ) was performed with the response to time to ignition (TTI). An appropriate post hoc test (Scheffe) was performed in case that the null hypothesis was rejected.

## 3. Results and discussion

### 3.1. Cone calorimeter

From the cone calorimeter test, the fire parameters of wood samples such as TTI, THR, PHRR, and TSP were measured and tabulated in Table 1. The radiative heat resistance of ZnO coated wood specimens was evaluated by the extent of TTI wherein the samples were exposed to different irradiation levels i.e., 20, 35 and 50 kWm<sup>-2</sup>. There was a clear increase in TTI for wood specimens coated with ZnO, across all tested heat fluxes and ZnO amounts. For instance, when 0.5 g ZnO was applied per square decimeter at a heat flux of 35 kWm<sup>-2</sup>, the TTI was found to increase by 26–33 % compared to the uncoated wood. The increase in TTI was even more significant for 1 g ZnO dm<sup>-2</sup>, ranging from 37 to 40 % (as shown in Table 1). These results suggest that applying ZnO to wood can function as a FR against radiative heat, thereby delaying the ignition of wood. The trend observed was that higher amount of ZnO led to greater increase in TTI. This is depicted schematically in Fig. 1, and the TTI values of the wood specimens are shown in Fig. 2 and summarized in Table 1. It is to be noted that in Sweden, the median response time of fire rescue services is ca. 7 min (Runefors, 2020), which is longer than the time to ignition observed in the current study. However, a longer time to ignition will slow the speed at which the flame spreads and thus, has implications towards human safety and building structural stability.

Fig. 3 depicts the heat release pattern and PHRR of the tested wood specimen. The trends in the HRR curves were different from those of TTI for each heat flux. At the heat flux of 20 kWm<sup>-2</sup>, the influence of ZnO on reducing HRR is completely negligible. However, at the higher heat fluxes, at 35 and 50 kWm<sup>-2</sup>, ZnO coating with 1 g.dm<sup>-2</sup> significantly lowers the HRR compared to that of the virgin wood. It is evident from the 2nd PHRR (Fig. 3) of the ZnO coating with 1 g.dm<sup>-2</sup>.

In general, the smoke production rate (SPR) curve for 20 kWm<sup>-2</sup> heat flux, seen in Fig. 3, shows a lower amount of smoke produced for both

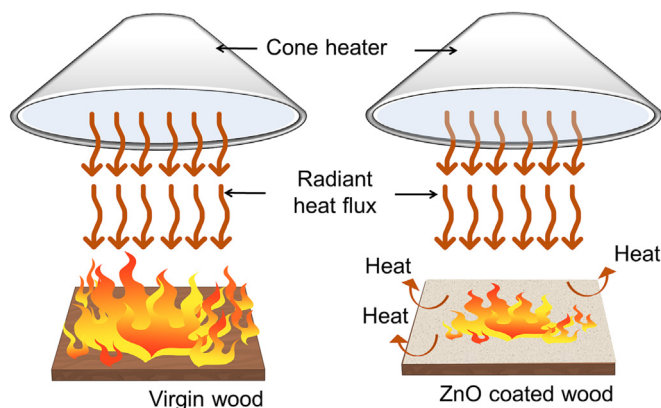


Fig. 1. Schematic representation of the behaviour of virgin wood and ZnO coated wood under radiant heat fluxes.

amounts of ZnO coatings. However, the effectiveness of the ZnO coating amounts, for a particular irradiation level, is difficult to elucidate. Especially for the first peak, before 200 s, the virgin wood and the ZnO coated (both the amounts) wood have similar smoke production rates. However, if the second SPR peak is considered, then, the wood with 1 g ZnO dm<sup>-2</sup> produced somewhat less smoke (especially for 35 and 50 kWm<sup>-2</sup> irradiance levels) than the other two specimen.

### 3.2. Surface morphology

The surface morphology of the wood specimens before and after the cone calorimeter test was studied by camera photography and the images can be seen in Fig. 4. It was found that when the wood caught fire, the ZnO layer remained intact while the wooden substrate developed cracks (see Fig. 4). Fig. 5 shows the surface morphology (observed through SEM) of ZnO-coated wood (1 g.dm<sup>-2</sup>) before and after testing in the cone calorimeter. The SEM images taken before and after combustion revealed that the morphology of ZnO did not change significantly. Although some ZnO particles agglomerated after being exposed to fire, they were still largely intact. However, the test results may have been influenced by the size of the particles. In order to determine the extent of this influence, further tests would need to be conducted using different particle sizes of ZnO. The

Table 1

The PHRR and TTI measured at different heat fluxes. For each test setup, three samples were performed.

Samples	PHRR [kWm <sup>-2</sup> ]	TTI [s]	Improvement in TTI [s]	Improvement for TTI in percent
<b>Heat flux of 20 kWm<sup>-2</sup></b>				
Virgin wood	158 ± 8	80 ± 8	-	-
0.5 g.dm <sup>-2</sup> ZnO	183 ± 14	106 ± 14	26	33 %
1 g.dm <sup>-2</sup> ZnO	168 ± 3	111 ± 8	31	39 %
<b>Heat flux of 35 kWm<sup>-2</sup></b>				
Virgin wood	203 ± 7	19 ± 3	-	-
0.5 g.dm <sup>-2</sup> ZnO	202 ± 8	24 ± 4	5	26 %
1 g.dm <sup>-2</sup> ZnO	194 ± 24	26 ± 3	7	37 %
<b>Heat flux of 50 kWm<sup>-2</sup></b>				
Virgin wood	254 ± 17	10 ± 2	-	-
0.5 g.dm <sup>-2</sup> ZnO	244 ± 33	13 ± 3	3	30 %
1 g.dm <sup>-2</sup> ZnO	227 ± 24	14 ± 2	4	40 %

See Appendix A for the ANOVA and post hoc analysis.

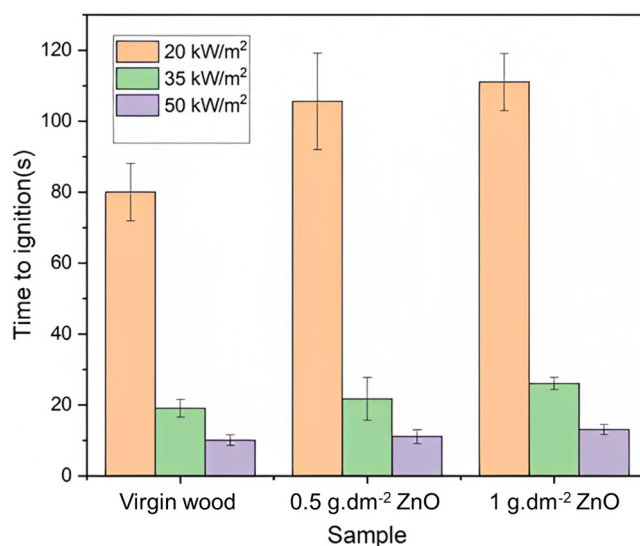


Fig. 2. Time to ignition (TTI) values of virgin and ZnO coated wood specimens at different heat fluxes.

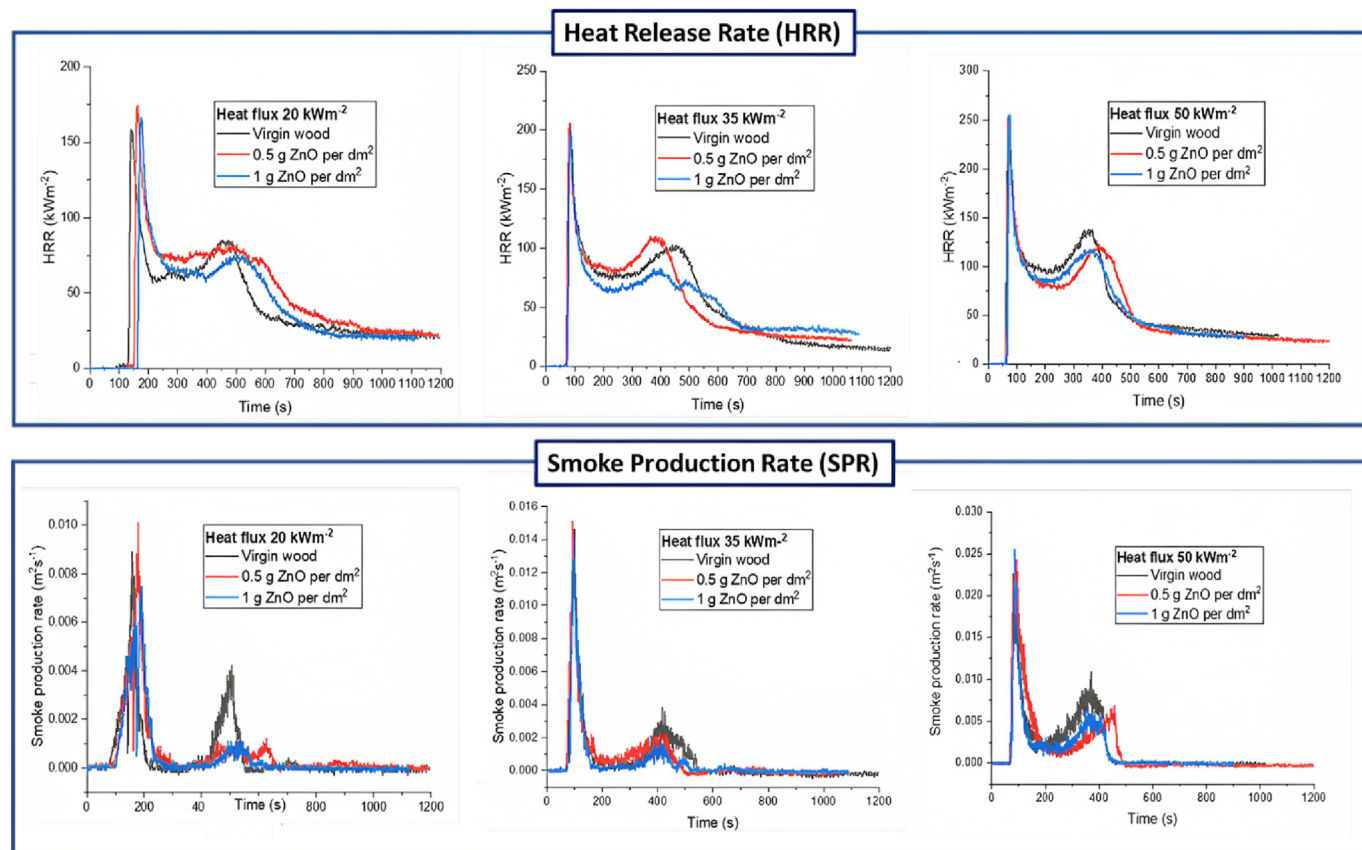


Fig. 3. Heat release rate (HRR) vs. time and smoke production rate (SPR) vs. time graphs of ZnO coated wood specimens for various heat fluxes.

ZnO particles appeared to have partially fused together after exposure to fire. This suggests the need for further investigation into whether this phenomenon can be leveraged to provide more coverage for the wooden surface.

#### 4. Conclusions

The aim of this research was to investigate whether ZnO powder could enhance the fire resistance of wood, particularly with respect to the time to ignition (TTI). The study found that the increase in TTI was consistent across all three heat flux levels tested, which were 20, 35, and 50 kWm<sup>-2</sup>. These results indicate that the addition of ZnO powder can significantly improve the fire safety of wooden surfaces by delaying ignition, and thus lowering the speed of flame spread. Adding just 0.5 g of ZnO dm<sup>-2</sup> resulted in a TTI increase of approximately 26–33 % compared to untreated wood. The 0.5 g ZnO dm<sup>-2</sup> coating did not show any effect on HRR, THR, and TSP values compared to the virgin wood. However, 1 g ZnO dm<sup>-2</sup> coating has shown lower HRR, THR, and TSP compared to the virgin wood at radiant heat fluxes of 35 and 50 kWm<sup>-2</sup>. The findings of this research provide strong evidence that ZnO has the ability to enhance the fire safety of wooden structures by effectively prolonging the time until ignition, which will consequently lower flame spread and the extent of burning. This will enable rescue and evacuation services to deal with a less severe fire, and potentially save lives and ensure structural stability. However, it is also important to make a coating formulation that holds ZnO particles covalently in such a way as to avoid leaching out of ZnO over the coating's ageing process. Surface functionalisation of ZnO with covalently linkable groups could be the choice in this regard and can form the basis for future research in this area. Additionally, the environmental fate of micron-sized ZnO after their usage in building fire-safety should be investigated in future research.

#### CRediT authorship contribution statement

*Olina Öhrn*: Roles/Writing - original draft; Formal analysis; Investigation; Conducting experiments.

*Kesavarao Sykam*: Roles/Writing - original draft.

*Sidique Gawusu*: Roles/Writing - review & editing.

*Rhoda Afriyie Mensah*: Roles/Writing - original draft; Software; Validation; review & editing.

*Lin Jiang, Qiang Xu, Ágoston Restás & Michael Försth*: Roles/Writing - review & editing.

*Vigneshwaran Shanmugam*: Roles/Writing - original draft; Writing - review & editing.

*Karthik Babu NB*: Roles/Writing - review & editing.

*Oisik Das*: Conceptualization; Data curation; Formal analysis; Investigation; Methodology; Project administration; Resources; Software; Supervision; Validation; Visualization; Roles/Writing - original draft; Writing - review & editing.

#### Data availability

Data will be made available on request.

#### Declaration of competing interest

All authors declare no financial/commercial conflicts of interest, with any parties.

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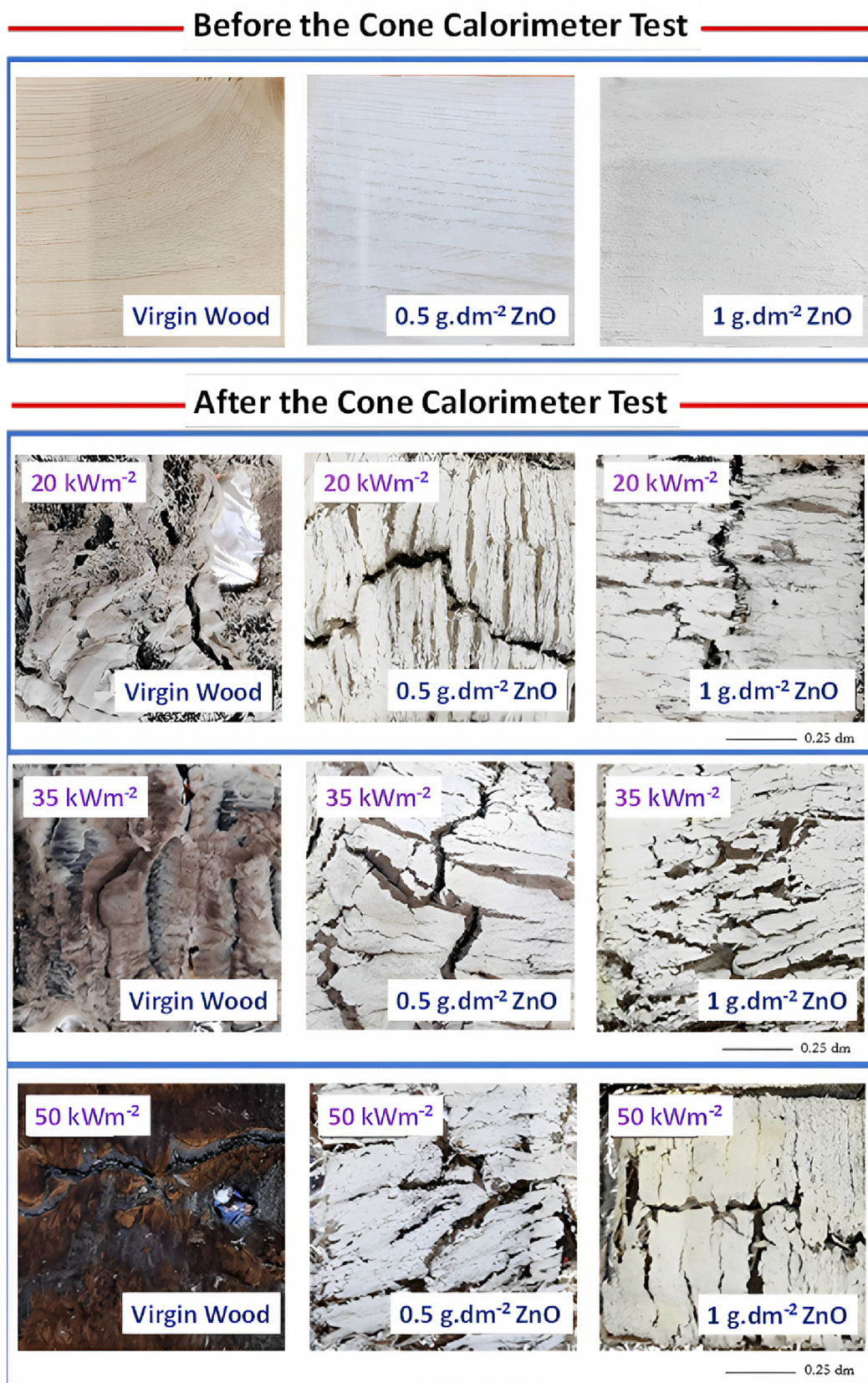


Fig. 4. Images of the samples before and after combustion at various heat fluxes.

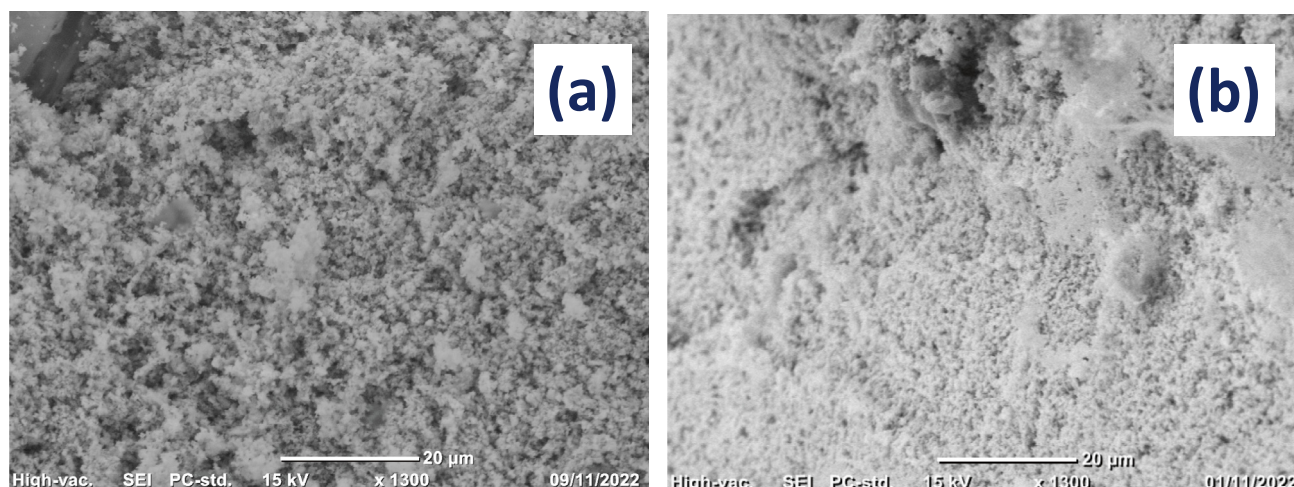


Fig. 5. SEM image of wood piece with 1 g·dm<sup>-2</sup> of ZnO (a) before and (b) after testing in the cone calorimeter at an irradiation level of 20 kWm<sup>-2</sup>.

Appendix A. ANOVA and post hoc test for time to ignition (TTI)

Source of variation	SS	df	MS	F	P-Value	F crit
Between Groups	41,865.85	8	5233.231	134.6971	0.000	2.510158
Within Groups	699.3333	18	38.85185			
Total	42,565.19	26				

Since F > Fcrit, the null hypothesis was rejected. The TTI means of the samples are not all equal. At least one of the means is different. Therefore, the following post hoc test (Scheffe) is conducted to identify where the difference lies. Note that coloured cells have significant mean differences.

Post Hoc	20 kW.m <sup>-2</sup> ; Virgin wood	20 kW.m <sup>-2</sup> ; 0.5 g.dm <sup>-2</sup>	20 kW.m <sup>-2</sup> ; 1 g.dm <sup>-2</sup>	35 kW.m <sup>-2</sup> ; Virgin wood	35 kW.m <sup>-2</sup> ; 0.5 g.dm <sup>-2</sup>	35 kW.m <sup>-2</sup> ; 1 g.dm <sup>-2</sup>	50 kW.m <sup>-2</sup> ; Virgin wood	50 kW.m <sup>-2</sup> ; 0.5 g.dm <sup>-2</sup>
20 kW.m <sup>-2</sup> ; 0.5 g.dm <sup>-2</sup>	25.6							
20 kW.m <sup>-2</sup> ; 1 g.dm <sup>-2</sup>	31	5.3						
35 kW.m <sup>-2</sup> ; Virgin wood	61.3	87	92.3					
35 kW.m <sup>-2</sup> ; 0.5 g.dm <sup>-2</sup>	55.6	81.3	86.6	5.6				
35 kW.m <sup>-2</sup> ; 1 g.dm <sup>-2</sup>	54	79.6	85	7.3	1.6			
50 kW.m <sup>-2</sup> ; Virgin wood	70.3	96	101.3	9	14.6	16.3		
50 kW.m <sup>-2</sup> ; 0.5 g.dm <sup>-2</sup>	67	92.6	98	5.6	11.3	13	3.3	
50 kW.m <sup>-2</sup> ; 1 g.dm <sup>-2</sup>	65.6	91.3	96.6	4.3	10	11.6	4.6	1.3

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