Thermal behavior and toxic emissions of flame retarded timber in fire enclosure tests

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Abstract

Timber in different forms contributes as first and secondary ignited material to the initiation and spreading of fires in industrial buildings. The aim of this work is to investigate experimentally the fire behavior of wooden surfaces treated or not with flame retardants in a 1.57 m³ fire enclosure linked to the FTIR analyzer in well ventilated conditions (75kg/h) that are usually encountered in industrial activities when large metal doors, ramps, ventilation openings, etc are open in order to serve the process of production. In order to simulate the "worst reasonable cases" of fire scenarios, wooden cribs have been constructed with complex geometry and configuration, and various quantities (grams) of ethanol were used as ignition sources. Seven (7) wooden crib fires were investigated using untreated pine wooden cribs or those treated at different percentages (%) of the total surface area with a water-based, flame retardant intumescent, suitable for internal surfaces. In most fully treated (100% F.R.) cases, even in a halftreated (50% F.R.) case, lower or almost equal to unity emissions were measured compared with the bare samples. This can be explained, in such cases, due to the fact that during the intumescent action there was either 'no ignition' of the samples (100% F.R.-treated cases), or a considerable ignition delay (50% F.R.treated case). Excessive HCN and NOx occurred in 60% of untreated cases due to the considerable involvement of the flame retardant paint in flaming combustion, since it contains N in its chemical composition.

It is proposed that the application of intumescent flame retardants on wooden surfaces located close to ignition sources in the most probable areas for a fire to break out, could be a safe and effective approach in reducing fire losses in industries

Keywords: wood, fire enclosure, flame retardant, heat release rate, ignition, FTIR analyzer, emissions.



1 Introduction

Wood has many good properties as a material. It is heavily used for construction and building because of its ease of processing, physical and mechanical properties, aesthetic, environmental and health aspects [1, 2]. Wood is also used in many industrial applications for purposes such as surface lining material, furniture, flooring, roof, shelves, pallets, wooden scaffolding for both offshore and onshore and packing cases [3, 4]. However with current regulations and standards [5], this is not allowed, as this would significantly add to the fire load within a room in a fire scenario, i.e. wooden materials in different forms, with the highest percentages, contribute as first ignited materials in fire initiation and spreading and have a major impact in fire losses [3, 4, 6].

All the above clearly signal the importance of controlling the ignition resistance and flammability of timber as used in different forms in various constructions. So, it is possible to increase the fire performance of wood, with the application of Flame Retardants. With the addition of a flame retardant, such as an impregnation treatment and surface coatings (possibly intumescent), the classification group of wood can be raised to class 1. Building Regulations class 0 can be achieved through the application of both types of flame retardant [3]. Thus, it may be possible to use wood as a surface lining material, if it is treated correctly.

There has been a significant but limited amount of work concerning flame retardants for wooden surfaces. Previous research has shown that flame retardants applied to wood have had a positive effect on the burning behaviour of wood, in terms of ignition and of the most important variable to describe fire hazard the heat release rate, H.R.R. [7]. Full scale fire experimental work by T.J. Shields et al. [8] compares flame retarded and non-flame retarded wood based wall linings, with flame retarded linings, delaying the ignition by some 11min 45s, although once ignited flame spread was very rapid. Intumescent Flame Retardants have also been the subject of study, in their own right. Wladyka-Przyblak and Kozlowski [9] evaluate the thermal characteristics of different intumescent coatings and found that, with regards to fire retardancy and heatinsulating properties, the most efficient coating was based on Amino resin, prepared from Urea, Dicyandiamide, Monoammonium Phosphate and Dextrin. Ostman and Lazaros [2] presented experimental data between fire retardant treated and untreated wood products. The test results show a significant differences between these two groups. The parameters included in the comparison are t_{ig}, HRR (peak and average values) and total HRR. The woodbased products were tested in different small-scale national fire tests and in the full-scale room fire tests. Fire retardant wood products achieve an improved classification both in present national systems and in possible new systems based on the cone calorimeter and room fire test.

Tsatsoulas et al. [6] used FTIR to investigate toxic emissions of eight species of wood. 'Significant' acrolein peak values are measured for all samples. At low irradiance (i.e., 35kW/m²), facing types of timber, e.g., MDF, Chipboard, with melamine or maple increases significantly the ignition resistance of MDF and

Chipboard by a factor of 1.5 to 2, due to the flame retarding properties of melamine and maple.

In the present work the effect of a typical water-based intumescent flame retardant (latest technology) on the common type of timber (Pine) was examined in medium scale (1m³ enclosed fire rig) experiments combined with online effluent gas analysis equipment (FTIR). Analysis involved thermal behavior, and toxic species analysis of the samples. The experiments were run under controlled airflow conditions. The airflow rate used in the experiment was 75 kg/h, i.e., corresponds to well ventilated fire conditions. The reasons for choosing the above-mentioned high ventilation rate were: (i) to reasonably simulate the ventilation conditions in the beginning of most real industrial fires, because it results from the processing of statistical data [4] the largest percentage of industrial fires have broken out in the middle of normal working days, when there is particularly increased activity of most industries, especially in the production areas. During these periods of time, all openings, e.g., large metal doors, ramps, ventilation openings etc., are open, in order to serve the production process, thus the ventilation conditions of these areas are not restricted. (ii) To allow comparisons through toxic yields (g/kg) estimations with small-scale experimental works in cone calorimeter [4, 6], where the experimental conditions are also well ventilated.

2 Experimental techniques

A 1.56 m³ enclosed fire test facility, 1.4m x 0.92m x 1.22m, was used with separate entrained air inlet at floor level and fire product exit at ceiling level [10] The enclosure had 25mm thick high temperature insulated walls attached to a steel backing wall that was air sealed. The enclosure had a high temperature air sealed glass door, which could be used to observe the early stages of a fire. This door was fitted with an outer door of 25mm thick insulation so that when the fire was fully developed there was no radiative heat loss through the glass door. The test facility was 1.4m, from the front glass window to the rear wall and smoke logging occurred when there was no depth of vision through the window. The fire load was placed on a central platform supported on three legs that went through to three load cells in the air plenum below the fire test chamber. This kept the load cells cool and allowed the fire mass burn rate to be determined through the fire. The air was fed from a compressor through a thermal mass flow meter into the fire enclosure through a plenum chamber under the fire enclosure floor, with air admission to the fire chamber through four slots on the floor at the base of each wall. The enclosure ceiling was instrumented with an array of Type K 3mm outer diameter mineral insulated exposed junction thermocouples. These were placed 70mm from the ceiling and the mean temperature was taken as the mean fire temperature. The fire product gases flowed across the ceiling and were exhausted from the fire through four slots around the periphery. The products flowed along the backside of the ceiling and out through a 152mm short flue into a cowl and then with air entrainment into a discharge chimney with extraction fan.

It was assumed that the fire product gases were well mixed at the 152mm flue and gas samples for toxic gases were taken through uncooled stainless steel tubes into 190°C heated sample lines, these were positioned before any dilution of the exit gases occurred in the discharge duct. Two sample probes were used simultaneously, both with 190°C heated sample lines. One was fed via a heated filter and pump to a conventional emissions analysis system with heated chemiluminescence NOx analyzer and to a paramagnetic oxygen analyzer. The second heated sample line was linked to a TEMET GASMET CR-Series portable FTIR .This has a multi-pass, gold-coated sample cell with a 2m path length and volume of 0.221. A liquid nitrogen cooled MCT detector was used that scans 10 spectra per second and several scans are used to produce a time-averaged spectrum. The signal to noise ratio is improved for longer signal averaging times. For fire research that develop slowly over several minutes, an overall response time of 5s was used and this was more than adequate to resolve the time dependent toxic gas production in the present fires which lasted for typically 20 minutes. The Temet FTIR gives a 2ppm resolution with an accuracy of 2% and a precision that is 0.01% of the measurement range. The whole detector cell was heated as well as the sample line, pump and filter. The analysis loses no species through condensations so that high MW hydrocarbons can be detected. The instrument was calibrated by the manufacturers using reference gas concentrations. The FTIR was calibrated for all the significant species that were present in the sample. The only calibration necessary prior to the test was to zero the instrument on nitrogen.

3 Experimental fires configuration

Tests were conducted in the enclosed Fire Rig using: I. Untreated wooden crib II. Fully flame retarded wooden cribs III. Partial flame retarded wooden crib. From the various types of wood found in different structures in the industry, pine was selected for medium-scale experimental investigation, since it is one of the most commonly used type of wood is "easy-to-use" and produced in large quantities, especially, from the Mediterranean forests [4]. It was decided to test the wood in form of cribs, because, in real fires there are complex wooden geometries and configurations strongly affecting the "spreading of fires" [5]. Thus, a wooden crib, which includes a crossed layer of sticks, simulates complex wooden structures, where the confinement of heat and cross-radiation among the surfaces allows for the efficient burning of wooden surfaces, and the rapid development of fires.

Wooden cribs with average fire load equal to 45 kg/m²(~765kJ/m²) were chosen. This represents the "worst reasonable case" of fire load among the wooden surfaces, which have contributed to the initiation and spreading of industrial Fires [4]. A total of 42 pieces of square faced 18mm x 18mm x 200mm were used comprising of ten layers of four pieces, with each alternate layer placed in the opposite direction. Then there were two pieces that were put on the bottom of the crib to act as feet to stabilize the crib. The untreated crib was weighed on a balance and was found to be of mass 1.7 kg with total surface area



0.7 m².In order for the crib to be easily ignited a small volume of ethanol was used in a dish. The ethanol was placed directly below the center of the crib (as "worst" case position) and was ignited using a blowlamp and the crib placed on top. A mass of 6 grams was used. This was deemed to be a suitable mass that would be sufficient to ignite the untreated crib and a small enough amount so as not to adversely affect the heat output and mass loss route, as the fuel would burn out quickly.

'Zero-flame' retarded paint was chosen for coating the wooden crib, since it is water-based and especially designed for interior wooden surfaces, like those simulating the wooden cribs under experimental testing. The 'Zero-flame' retarded paint was applied on six (6) wooden cribs, as detailed below. A paintbrush was used to apply in two (2) coats with drying time between the coats 18 hours. The following (% flame-retarded % bare) wooden cribs were tested experimentally in order to examine different cases of fire development at the ventilation rate mentioned above.

- 100% flame retarded, where all their surfaces were painted with 'Zero-Flame' retarded paint. Total surface, coated in two (2) layers of flame retarded paint: 0.7 m². Overall, three (3) wooden crib tests were performed with 100% flame retarded surfaces. For their quick ignition, 6g ethanol, 20g ethanol, and 30g ethanol were used, respectively, in the manner, described in the case of ignition of virgin crib. The reason for conducting three different tests using 100% flame retarded crib with three different quantities of ethanol as ignition sources, was to simulate different power of real ignition sources [4].
- 50% flame retarded, 50% bare wood, where the first 5 layers (21 sticks) were treated with 'Zero Flame' retarded paint(two(2) coats), and the rest 5 layers (21 sticks) were left untreated. One (1) test was carried out, and 6g ethanol was used as ignition source in the same way described before.

Table 1: Wooden crib tests. The (%) indicates the fraction of the crib (starting from the bottom, which was either flame retarded or bare).

Fuel	Condition of the fuel surface	Flow rate (kg/h)	Fuel size(cm)	Fuel Quantity (g)	Ignition source
Pine	Untreated	75	20x20,4sticks per layer, 10 layers high	1,785	6g.ethanol
As above	100%F.R	75	As above	2,025	6g.ethanol
As above	100%F.R.	75	As above	2,018	20g.ethanol
As above	100%F.R	75	As above	2,031	30g.ethanol
As above	50% Treated	75	As above	1,912	6g.ethanol
As above	60%Untreated	75	As above	1,875	6g.ethanol
As above	60%Untreated	75	As above	1,881	20g.ethanol
Total: Seven(7)tests					

• 60% bare wood, 40% flame retarded wood, where 0.437m² were left untreated, i.e. 26 sticks in the first 7 layers, while the remaining 0.268 m², i.e., 16 sticks, were painted with flame retardant paint(two(2) coats), included in the 7th and 10th (last) layer of wooden crib. Two (2) tests were performed with 6g and 20g ethanol as ignition source, respectively. The reasons for using the above two (2) quantities as ignition sources were: to simulate sources of real fires starting, guided by the statistical analysis of industrial fires [4], and show whether they affect the faster spreading of fires or not.

All the above tests using various wooden cribs with different strength of ignition sources give an indication whether the application of flame-retardants at different parts of wooden surfaces located near potential ignition sources (as first or second ignited materials) contribute to suppression or to slower fire developing, and show how they may affect the concentrations of the released toxic gases when the wooden surfaces are burned.

4 Experimental results

4.1 Fire development results

Test results cover (i). HRR (kW/m²) (ii) Mass Loss Rate (kg/s) (iii) Ceiling Temperatures (C°). It is reminded that % indicates the fraction of the crib (starting from the bottom) that was either flame retarded or untreated. One untreated sample was tested using 6g of ethanol as ignition source. The corresponding plot in Figure 1 shows that the untreated sample clearly burned faster. The presence of the FR paint clearly suppressed (either partially or totally) the combustion process. 'No ignition' of the fully treated (100%) crib was detected even when the ignition source was changed from 6g to 30g of ethanol.

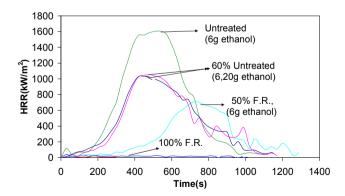


Figure 1: HRR(kW/m²) vs. time for pine cribs with different FR treatment levels with 75kg/h air flow rate.



A "weak" flame initially developed from the burning of ethanol, which "triggered" the in-tumescent flame retardant paint to expand and form "instant firewalls" to contain and finally suppress the developing fire. Thus, at the end of the experimental process (1200 sec), the flame retarded cribs of the various cases remain almost "intact". The intumescent action is only seen on the surface of the sticks in the near vicinity of the initial flame from the ignition of ethanol. Fires exhibited two phases, flaming combustion and char smoldering. Considering the tests shown in Figure 1 flaming combustion ends up at about 900s. The fire mass loss as a percentage of the initial mass is shown as function of time in Figure 2. It is obvious that the fire mass loss of flame retarded samples is slower, especially when the treated surfaces are closer to ignition source.

A direct comparison of the effect of the flame retardant is achieved by 'Peak Heat Release Rate Reduction Factor':

$$'PHRR.R.F.' = \frac{PHRRCoatedSample}{PHRRBareSample}, \qquad (1)$$

The peak HRR values reported in Figure 3 for 100% treated samples are the greatest values measured although they did not necessarily form a well-defined 'peak'.

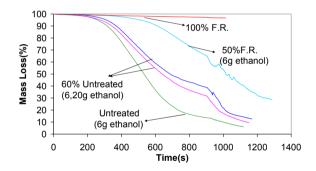


Figure 2: Mass loss (%) vs. time for pine cribs with different FR treatment levels with 75kg/h air flow rate.

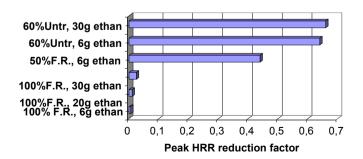


Figure 3: Comparative effects in peak HRR of flame retardant coatings.



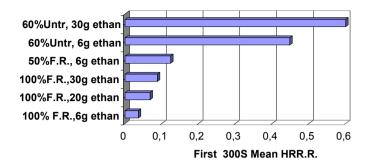


Figure 4: Comparative effects in first 300s mean HRR of flame retardant coatings.

Another measurement of the effectiveness of the FR treatment is the average HRR over the first 300s divided by the equivalent HRR of the untreated crib (eqn(2)) shown in Figure 4. This is achieved by defining:

'First300sMeanHRRRatio' =
$$\frac{300sMeanHRRCoatedSamples}{300sMeanHRRBareSamples},$$
 (2)

An interesting finding evident in Figure 3 is that with regard to the peak HRR the 50% treated test produces about 45% of the untreated peak HRR. However, the effectiveness of this partial treatment is in fact far greater when viewed in terms of the '300s average HRR' (see Figure 4), where it is shown that it is almost as effective as the 100% treated sample (generally less than 15% of the average HRR of the untreated sample). The results clearly show that FR treatment can be very effective and additionally it is not necessary for the whole area to be treated. A partial treatment of the areas nearest to potential ignition sources may be a workable solution which can almost be as effective as the 100% treatment but absolutely much more economical.

In the case of the bare sample, the peak fire temperature reached was 515° C. In all cases of 100% F.R. samples very low temperatures developed 41° to 45° . In both cases of 60% untreated peak temperatures are about 390° i.e. lower by a factor of 0.75 than of untreated sample. In the case of 50% FR, peak temperature is 290° (at 872sec) which is lower than the corresponding values of 60% untreated samples by a factor 0.75.

Published data on the burning characteristics of intumescent coated timber using medium or full-scale experiments are limited. Birgit et al. [2] used a full-scale standard room to test untreated and treated with flame retardants wood based products. They concluded that the time to flash-over for the FR. wood products was much longer than for the untreated wood products of the same type. An ignition source of 100kW was initially used. In the case of flame retarded samples it was raised to at least 300kW for flash over to occur.



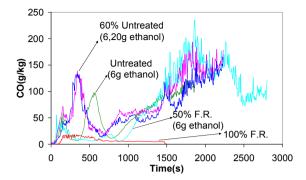


Figure 5: CO mass emissions (g/kg) vs. time(s) for various (pine) cribs with 75kg/h air flow rate.

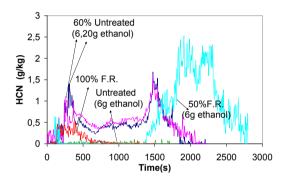


Figure 6: HCN mass emissions(g/kg) vs. time(s) for various (pine) cribs with 75 kg/h air flow rate.

4.2 FTIR toxic gas analysis

FTIR analyzer was connected to the enclosed fire rig for all the tests. Only measurements of toxic gases that show "significant" concentrations for life safety based on the maximum exposure levels recommended by COSHH for individual combustion products [10, 11] are reported in this work. Toxic yields of main toxic gases were assessed (see Figures 5 and 6) for better comparison of toxic species of wooden samples, untreated or treated in different parts with typical flame retardant, and finally to evaluate the overall toxicity [4].

A direct comparison of the effect of the flame retardant used is achieved by 'Peak n_(toxic gas) Coefficient Ratio' (P. n _(toxic gas). R.) (see eqn. (3) below). This coefficient ratio can be applied only during flaming combustion peak values, as this phase is more important in the present work, which is interested in examining the development of fires on wooden samples painted or not with fire-retardant paints during the early stage of fire development, and for a period of time up to 15 min, which, in real fire conditions, covers the time needed for the



evacuation of the industrial plant by its staff, the potential intervention of the fire-safety staff of the plant, and the arrival of the fire department to extinguish the fire.

$$\text{`P.n.}_{(toxicgas)}.\text{R.'} = \frac{P.n_{toxicgas}.CoatedSample}{P.n_{toxicgas}.BareSample},$$
(3)

where n $_{toxic\ gas}$ volumetric (ppm) or mass (g/kg)) emissions. An alternative way of assessment of the effectiveness of the Flame Retardant treatment is the average n $_{toxic\ gas}$ (ppm or g/kg) over the first 300s divided by the equivalent n $_{toxic\ gas}$ (ppm or g/kg) of the untreated crib (see Eqn.4),

'First300sMeann_(toxicgas).R.'=
$$\frac{300sMean \text{ n}_{toxic gas}\text{CoatedSample}}{300sMean \text{ n}_{toxic gas}\text{BareSample}},$$
 (4)

Therefore, the effects of flame retardant treatment on major toxic emissions compared with the bare samples are shown on the following Table 2.

Table 2: Comparative effects of flame retardant treatment on major exhaust emissions (during flaming combustion).

Coated emission	100%F.R. 6g	100%F.R. 20g	100%F.R. 30g ethanol	50%F.R. 6g ethanol	60% Untreated 6g	60% Untreated 20g
Bare emission	ethanol	ethanol	etnanoi	etnanoi	ethanol	ethanol
'Peak CO(ppm) Ratio'						ı,
'Peak CO(g/kg) Ratio'			-		1	1
'First 300sMean CO(ppm) Ratio'	↓	↓	≈	↓	1	1
'First 300sMean CO(g/kg) Ratio'	↓	≈	≈	≈	1	1
'Peak HCN(ppm) Ratio'		!!	↓	1	11	11
'Peak HCN(g/kg) Ratio'	*	*	~	*	11	
'First 300sMean HCN(ppm) Ratio'	*	*	~	↓	11	11
'First 300sMean HCN(g/kg) Ratio'	*	*	≈	*	111	111
'Peak N0x (ppm) Ratio'	↓	1	*	*	11	11
'Peak N0x (g/kg) Ratio'		1			11	11
'First 300sMean NOx(ppm) Ratio	1	1	1	1	111	11 11
'First 300sMean NOx(g/kg) Ratio'	11	11	11	11	111	111

Each arrow / indicates decreasing/increasing up to a factor of three. Two arrows together is equivalent to a change by a factor of 3-6. Three arrows together is equivalent to a change by a factor for greater than 6.

In most fully treated (100%) cases, even in the half-treated (50%) cases, lower or almost equal to unity emissions were measured compared with the bare samples. This is due to the fact that, in such cases, due to the in-tumescent action, there was either 'no ignition' of the samples (100%-treated cases), or a considerable delay was seen (50%-treated cases). It is worth noting that 50% treatment is some cases was more effective to reduce toxic emissions compared with 100%-treated cases and, of course, much more inexpensive. This may be attributed to the fact that less flame retardant paint was involved in combustion in these cases. Excessive HCN and N0x occurred in 60% of the untreated cases due to the considerable involvement of the flame retardant paint in flaming combustion, since it contains N in its chemical composition, as mentioned before.

It will be apparent in the results that mass emissions (g/kg) are observed to significant increase over untreated species in a number of occasions compared to volumetric emissions (ppm). The reason is that the combustion of the untreated sample is more efficient; thus, a total mass is burnt significantly reduced with the treated samples. The mass of untreated sample burnt is in the denominator of the yield fraction [4, 6] thus the fraction is increasing.

5 Conclusions

As regards the Medium-Scale assessment of treated samples, the most widespread type of wood was chosen for testing in the form of wooden crib in a Fire Enclosure, using a different power of ignition sources, thus simulating real cases of fires. The main findings are the following:

- In all fully treated (100%) cases, there was no ignition, and increasing amounts of ethanol, i.e., 6, 20, and 30g, were used as ignition sources. In half-treated (50%) cases, there was a considerable ignition delay (> 300 sec), as well as a reduction in peak HRR values by a factor of 2.
- No significant differences were observed at the time to ignition compared with the untreated cases, in 60% of untreated cases, along with a reduction of the peak HRR values by a factor of 1.3.
- Lower values of toxic emissions or almost equal to unity are released in most fully treated (100%) cases, compared with the untreated cases. The half-treated (50%) cases released have similar or even lower values than fully treated cases, as seen in several cases. Increased values of toxic emissions, compared to untreated samples are observed in 60% of the untreated cases, due to higher involvement of the flame retardant paint in flaming combustion. Excessive toxic mass emission occurred in the latest cases.



Based on the above findings, it is proposed that the application of intumescent flame retardants on wooden surfaces located close to ignition sources in the most probable areas for a fire to break out, could be a safe and effective approach in reducing fire losses in industries.

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