



## Research Article

### Toxic Emissions from Processed Wood in Cone Calorimeter Tests

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#### ABSTRACT

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Despite the high number of deaths in fires due to toxic gases, there are no specific toxic gas requirements for materials used in buildings. The only standard for building materials in fire tests is smoke visibility obscuration. As our modern world increasingly uses wooden materials in buildings, a comprehensive understanding of their chemical nature and fire-related properties becomes indispensable. Oriented Strand Board (OSB), Medium Density Fiberboard (MDF), and Chipboard faced with white melamine (CFM) were investigated for toxic gas measurements. The tests were conducted using a standard cone calorimeter with raw gas sampling (predilution) via an 80mm diameter chimney mounted atop the cone heater exit. Heated gas sampling and Gasmeter FTIR toxic gas analysis were done using a 20-hole mean gas sample probe at the conical heater outlet plane. Each wood sample was exposed to the cone calorimeter radiating at 35 kW/m<sup>2</sup>, with ignition delays of 69 s (OSB), 142 s (CFM), and 54 s (MDF). Although the peak heat release rates (HRR) were similar across samples, the time variations differed, with CFM showing slower fire growth to peak HRR. The glued surface and manufacturing process introduced about 5% of organic nitrogen compounds into MDF and CFM, leading to very high levels of HCN immediately after the fire started. OSB contained 1/10<sup>th</sup> the organic nitrogen of CFM and MDF, leading to much lower HCN levels. However, OSB produced four times more formaldehyde and acrolein than CFM and MDF, dominating early-stage fire toxicity. The critical toxic species for all three wood types were carbon monoxide (CO), acrolein, formaldehyde, and benzene, based on both LC50 and COSHH15min basis, which indicates escape impairment.

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## INTRODUCTION

Over 60% of fire deaths in the UK and USA result from the inhalation of toxic effluents (Government, 2015). Smoke from fires contains toxic gas effluents and visible smoke is regulated for building fire products. Smoke reduces visibility and this may impair escape if the fire exit signs cannot be seen. However, the toxic gases in smoke contain irritant and acidic gases that attack the eyes and impair vision and cause respiratory problems. This slows the walking speed to an escape route or forces the occupants to go the wrong way as they cannot breathe. Irritant gases may thus impair escape, increasing the risk of a lethal exposure to asphyxiant gases such as CO, leading to the death of those trying to escape. The appalling number of deaths, in the Grenfell Towers fire in London on 14th June 2017, was mainly from toxic gases and fine particulates emitted in the fire that involved the external wall cladding at an early stage. Despite the significant number of deaths in fires due to toxic gas exposure, there are currently no regulations mandating toxic gas requirements for materials used in building construction. The only standard for building materials in fire tests is smoke visibility obscuration. This work compares the toxic gases produced from 3 different types of processed woods used for construction.

Wood and other cellulosic materials are extensively used in the construction of homes and other buildings (D.K. Shen, M.X. Fang *et al.* 2006) and wood is the dominant fire load in homes and other buildings (Persson and Simonson, 1998). Processed woods such as plywood, MDF, blockboard, some structural woods and laminated veneers are used in modern buildings for surface finishing, furniture, flooring, structural wood, scaffolding, ceilings, shelves and partitioning. These processed woods are manufactured with adhesives, resins, binders and other additives which contribute to the toxic gases released in the event of fire. It will be

shown in the present work that the binders release toxic products early in the fire, which is more hazardous than their release later in a fire. Wood might not necessarily be involved in the start of a fire, but it will contribute to the fire load and the toxicity once ignited.

The variability of the composition of wood makes the toxic products and smoke released variable. The thermal decomposition of wood products makes the toxic gas products complex, with few publications using heated FTIR (Fourier Transform Infrared) to determine the complex composition (Neviaser and Gann, 2004). Purser (Purser, 2010), has shown that the most important toxic products from fires are CO, HCN and irritant gases. But, HCN, HCL, and HBr are usually low except when the fire load contains organic N, Cl or Br fire retardant compounds (Purser, 2010) or there is a large organically bound fuel nitrogen content, as in the present work. These gases fall under the category of either asphyxiant gases, which the oxygen supply to the body tissue leading to loss of consciousness and death or the acidic and irritant gases which cause impairment of escape by irritating the eyes and the upper respiratory tract and also inflammation of the lungs (Purser and McAllister, 2016).

There is a lot of work in the literature on ignition, heat release and optical obscuration smoke emissions of wood using the cone calorimeter, but few on the toxicity of processed wood. Most publications on the cone calorimeter for toxic gas analysis sample from the dilute sampling point of the exhaust tunnel, where the concentration of the toxic gases is underpredicted due to post primary cone combustion oxidation in the secondary entrained air. The most extensive work on wood fire toxicity using multi-species heated FTIR analysis has been carried out by Andrews and co-workers (Aljumaiah, Andrews *et al.* 2010),(Aljumaiah, Alshammari *et al.* 2010). In

the first use of a calibrated heated FTIR with pine wood crib fires in a compartment Andrews *et al.* (Aljumaiah *et al.* 2010) showed that the peak toxicity that impacts on means of escape (Control of Substances Hazardous to Health (COSHH<sub>15min</sub>)) occurred 30mins. after the start of the fire, when the mean fire ceiling temperature was 250°C. 79% of this toxicity was due to acrolein and 18% to Formaldehyde. However, in the early stages of the fire after 2 mins., when temperature was lower at about 100°C the total toxicity N was 3 and three gases contributed to this: Formaldehyde (70%), Benzene (18%) and CO (12%). HCN was low in these tests with a peak at 15 ppm, which was not a significant contribution to the fire toxicity. This HCN peak occurred at 15 mins. into the fire and coincided with the peak temperature of 350°C and was formed from nitrogen in the air as part of the NO<sub>x</sub> formation kinetics. In the present work HCN levels of up to 1800 ppm 2-3mins after the start of the fire occurred and this is quite a different HCN source, from the N content of the resin material in the binders of the processed woods.

Pine wood crib fires in a 1.6 m<sup>3</sup> compartment fire were studied for a range of wood crib sizes and air ventilation rates by Mustafa *et al.* (Mustafa *et al.* 2015). They found that the main toxic gases at the peak toxicity time were Acrolein and Formaldehyde in terms of impairment of escape and CO, Formaldehyde and Acrolein for LC<sub>50</sub> toxicity. The peak toxicity occurred 22 – 30 minutes in the fire duration, which is quite different than in the present work where peak toxicity occurred immediately after the start of the fire. Alarifi *et al.* (2015) investigated large pine wood crib fires in a full-sized domestic house lounge with the door open. The peak HRR was 3MW and mean ceiling layer gases were analysed for toxicity, which showed that Acrolein, Formaldehyde and CO were the most important toxic gases, as was the case in the smaller pine wood crib fires.

Mustafa *et al.* (Mustafa *et al.* 2019) has used the present cone calorimeter methods to investigate the toxic emissions of pine wood for free ventilation. The development of the experimental methods were described by Irshad *et al.* (Irshad, 2017) and used a short chimney discharge to the cone calorimeter from which a mean gas sample was obtained. The results showed that the peak HRR was 200 kW/m<sup>2</sup> 300s after the onset of combustion. This was also close to the peak toxicity condition (LC<sub>50</sub> (Lethal Concentration)) and in the work of Mustafa the particle number was determined and shown to peak at the peak HRR condition. The present work shows faster flame development and quicker generation of the peak toxicity, relative to pine wood.

Tsatsoulas *et al.* (2009) investigated the thermal behaviour and toxic emissions of 8 different wood samples (mostly processed) but sampled the dilute gases on the FTIR, which tends to underestimate the concentration of gases released. They (Tsatsoulas *et al.* 2009) found significant amounts of acrolein released by all samples and higher emissions of CO, HCN and NH<sub>3</sub> for wood samples covered by Melamine.

There are several methods of assessing the toxicity of fire products. The most common is the LC<sub>50</sub> 30 min exposure concentration which aim at predicting the concentrations at which 50% of the people will die in the fire if exposed to the gas concentration for 30 minutes (Purser, 2010). European short term occupational exposure limits are detailed in the COSHH 15 minutes exposure regulations (EH40/2005. 2005), equivalent to the US short term AEGl2 10 minutes exposure limits (Council, 2015). The COSHH<sub>15 min</sub> toxic gas concentration represents a safe condition for 15 minutes where there will be no impairment of escape. The Acute Exposure Guideline Levels (AEGl) (Purser, 2010) are the USA guidelines having three different exposure levels: AEGl 1 for non-

disabling and are basically like the COSHH 8-hour levels; AEGL 2 are the COSHH<sub>15min</sub> levels equivalent for disabling and impairment of escape based on 10 minute exposure, and AEGL 3 for lethality which are similar to LC<sub>50</sub>.

The COSHH (EH40/2005 2005) limits are not common in fire toxicity, apart from the work of the University of Leeds toxicity research group, but they are the best set of data for assessing multi-component toxicity with a legally agreed methodology, which is comparable to the LC<sub>50</sub> N-gas model (Levin 1996; Babrauskas, *et al.* 1998). The authors have shown that COSHH<sub>15min</sub> and AEGL<sub>10min</sub> give similar results for when the most toxic condition for impairment of escape occurs. The COSHH<sub>15 min</sub> assessment methods of fire toxic gases and the LC<sub>50</sub> are used in this work to assess the toxicity of gases emitted from processed wood fires.

## MATERIALS AND METHODS

Three types of processed wood were investigated, and all are used in building construction and internal fittings in buildings: Oriented strand board (OSB), Chipboard faced with melamine (CFM) and Medium density fibreboard (MDF). The processed woods were cut into the standard cone calorimeter 100 mm x 100 mm size as shown in Fig. 1. The characteristics of the three processed wood materials are given in Table 1 and pictures of the three samples in the cone calorimeter sample holder are shown in Fig. 1. The Ultimate analysis to determine the water, volatiles, fixed carbon and ash content of the samples was determined using a Shimadzu TGA-50 with a TA60WS processor. Proximate analysis was carried out using the Thermo Flash EA Elemental analyser to determine the CHONS. The gross calorific value was determined using a Parr 6200 oxygen bomb calorimeter.

**Table1:** Fuel Properties

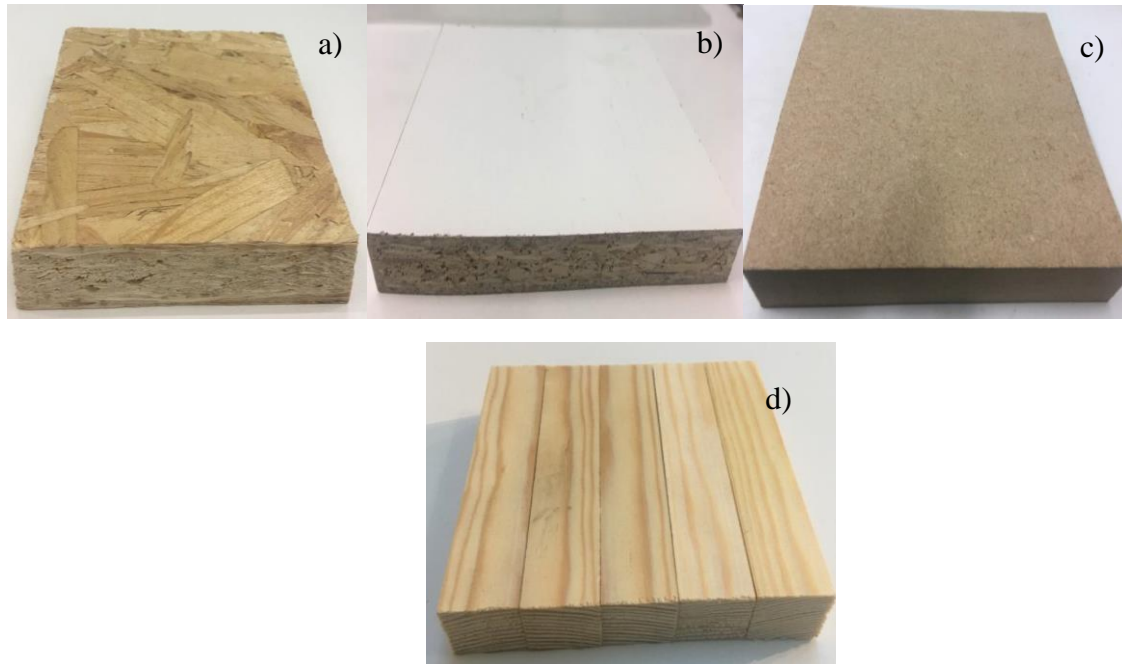
Parameter	OSB	CFM	MDF	PWS
Proximate Analysis (Wt. %) (daf)				
Volatile Matter	84.27	82.87	83.19	86.54
Fixed Carbon	15.73	17.13	16.81	13.46
Ultimate Analysis (Wt. %) (daf)				
Carbon	50.32	49.11	50.61	53.95
Hydrogen	6.36	6.50	6.80	6.79
Nitrogen	0.41	4.39	5.04	0.11
Sulphur	0.00	0.00	0.00	0.00
Oxygen	42.91	40.01	37.55	39.15
Stoichiometric A/F by carbon balance	5.38	5.17	5.63	5.89
Gross Calorific value (MJ/kg)	19.5	18.7	19.1	18.9
Moisture (as received) (%)	6.18	4.21	2.73	6.18
Ash (as received) (%)	0.74	4.14	4.45	2.27

Table 1 show that the organic nitrogen content of CFM and MDF was about 5%, which is

extremely high and will be shown to give rise to very high HCN toxic emissions shortly after the

start of the fire. The ash content of the CFM and MDF is also high and may indicate the presence of a fire retardant. The stoichiometric A/F was quite similar for all three processed woods and all had a similar volatile content of 83-84% and so a dramatically different fire on the cone

calorimeter was not expected. It will be shown that even with free ventilation the fire equivalence ratio at the exit from the cone heater was richer than stoichiometric in the early stages of the fire.

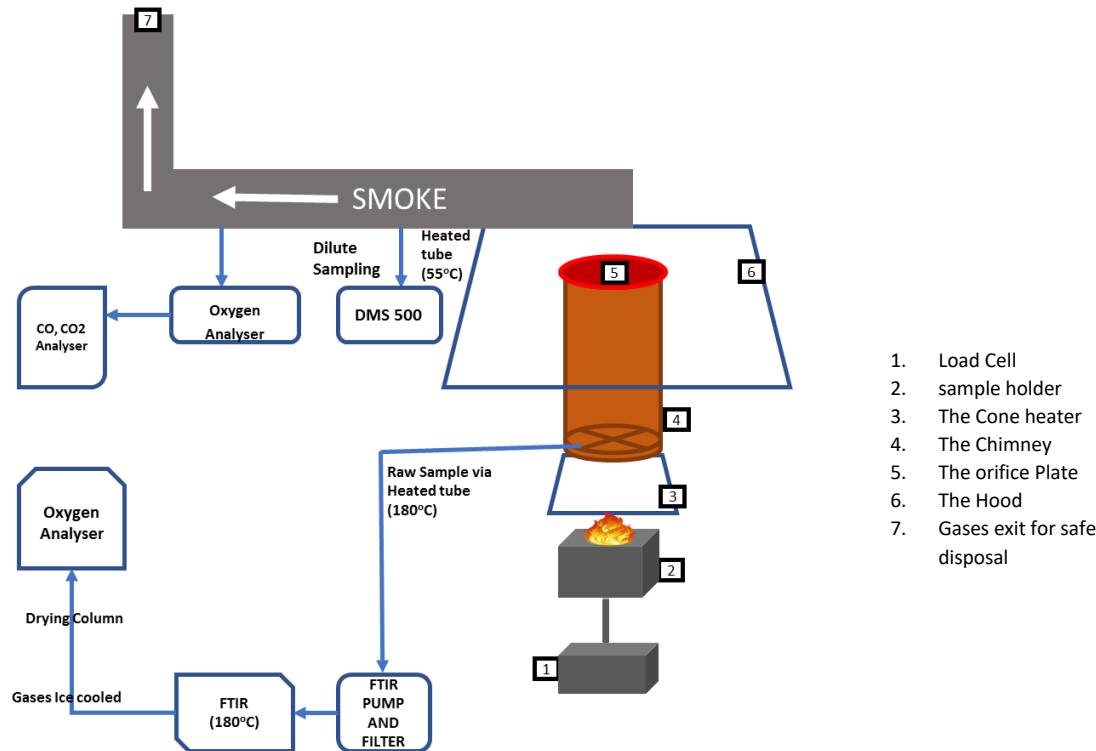


**Fig. 1:** (a) OSB b) Chipboard with Melamine c) MDF d) PWS

### The Experimental Procedure

The standard cone calorimeter (ISO 5660) designed to measure the heat release rate and flammability properties of materials were used for toxicity measurements. The cone calorimeter replicates the oxidative pyrolysis stage (class 1b) and well-ventilated flaming fires (class 2) classification of the ISO 19706 (19706:2011 2012). Irshad *et al.* (2017) have developed the standard cone calorimeter (ISO 5660) (5660-

1:2015 2015) to enable direct raw exhaust gas analysis from a chimney on top of the electrically heated cone exit. The cone can also be used to simulate compartment fires using the controlled atmosphere enclosure (Irshad, 2017) with a metered airflow, but it was used in the present work with the same chimney and mean gas sample arrangement as developed by Irshad *et al.* (2017). The cone calorimeter set up is shown in Fig. 2.



**Fig. 2:** The cone calorimeter

The wood samples were heated at  $35 \text{ kW/m}^2$  with free ventilation. This is the British Standard (5660-1:2015 2015) recommended heat flux for use in material evaluation for performance in fires. The processed wood samples had a production thickness of 18 mm (OSB), 15 mm (Chipboard with melamine) and 18 mm (MDF). The experiments were carried out in the horizontal orientation in a 100 x 100 mm square sample holder of the cone calorimeter, exposing the top surface to the applied radiant heat flux. The initial weight of the wood was determined by the load cell as 110 g (OSB), 95 g (CFM) and 103 g (MDF). The load cell was calibrated by checking with reference weights at the start of each test programme and was very stable. The  $35 \text{ kW/m}^2$  radiant heat flux caused the thermal decomposition of the samples leading to auto ignition of the evolved gases and the auto ignition delay time was determined in the tests at 69 s (OSB), 142 s (CFM) and 54 s (MDF). The fire continued until flaming combustion ceased

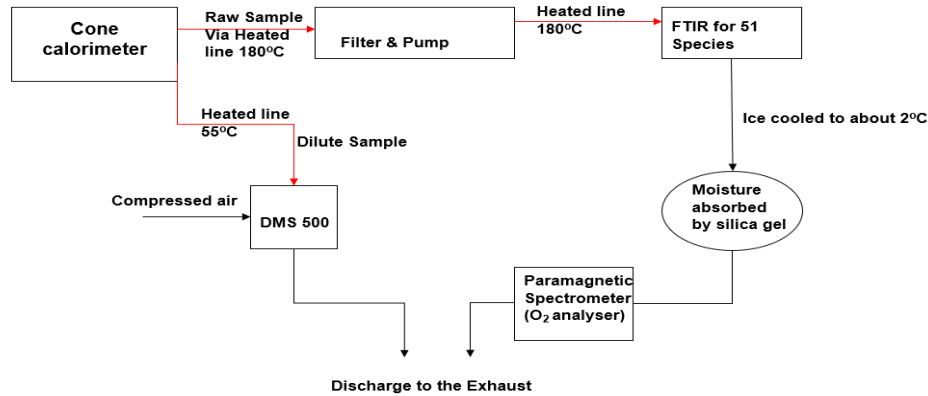
and there was only char burning. It will be shown that this transition from flaming combustion to char smouldering combustion was associated with a change in the release of toxic gases.

#### Sampling and Analysis Method

The cone calorimeter experimental setup was modified (Irshad, 2017) to enable a raw gas (predilution) sample to be obtained from the exit of the cone. A 20-hole X sample probe was mounted on top of the exit plane of the cone heater for the sampling of the mean composition of the raw gases. The X probe was mounted in a 76 mm diameter duct with flanges joining the cone heater exit to the chimney. This gas sample probe duct was 40 mm long. An 80 mm diameter chimney, the same diameter as the cone outlet, and 210 mm long was mounted on top of the sample probe and the total chimney length was 250 mm. To avoid the backflow of air down the chimney, a grid plate restrictor was placed at the chimney exit to increase the

exhaust flow pressure loss, which then prevented back flow of air down to the sample probe. This

orifice plate on the chimney had five 6.3 mm diameter holes.



**Fig. 3:** Schematic of the analyser

Fig. 3 shows the sample gas transport system to the gas analysers. The gases from the exhaust, chimney, were transported via a heated 180°C sample line to a heated pump (2.5-3 lpm) and filter. A second heated line was connected to the pump to transport the gases to the heated Gasmeter FTIR calibrated for 60 species including all the toxic gases that can be generated in fires. The raw gas sampling was used because it prevented any post oxidation of species by dilution of the gases with ambient air. It also prevented any losses of condensable gases that would occur if the exhaust was just thermally cooled (Alarifi, Phylaktou et al. 2015). The gas sample from the FTIR outlet was transported to a refrigeration cooler at about 2°C via a PTFE tube and then to a silica gel column to remove remaining water vapour, before going into a paramagnetic oxygen analyser, after which it was discharged through the cone calorimeter discharge duct.

### Toxicity Assessment

The toxicity of the gases produced by the fires was assessed using a modified FEC N-gas model 20. The N-gas was obtained by taking the ratio  $n$  of the concentration of all the toxic species

measured by the FTIR and dividing by either the LC50 values or the COSHH<sub>15 min</sub>. These ratios were summated to produce the total toxic gas  $N$  ratio. This  $N$  is a measure of the risk of deaths (LC<sub>50</sub>) or impairment of escape (COSHH<sub>15min</sub>) in fires.  $N$  is also a scalar parameter that indicates the level of smoke dilution necessary to make breathing the smoke not a death or means of escape hazard.

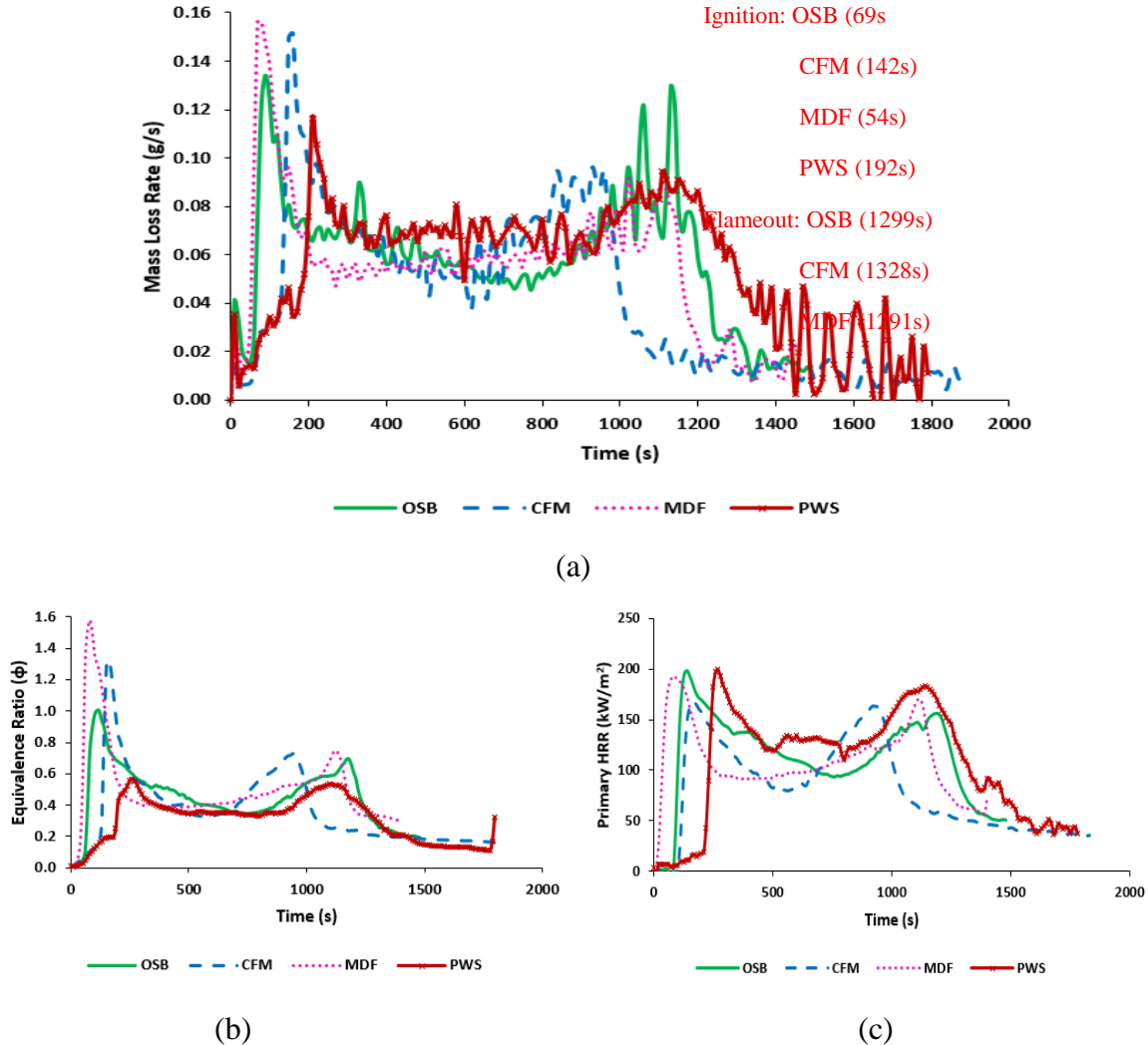
## RESULTS AND DISCUSSION

### Mass Loss, Equivalence Ratio and Heat Release Rates (HRR)

Figure 4 shows that the ignition delay was much shorter for the MDF fire, due to the richer mixtures during the delay period, as shown in Fig. 4b. The chipboard faced with melamine (CFM) had a prolonged ignition delay of 142 s compared with 54 and 69 s for MDF and OSB respectively. The melamine facing delayed the thermal decomposition of the processed wood. All the materials had a mass loss rate at steady state of 0.06 g/s with a much slower burn rate during the char burn phase from around 1100 s. Carbon balance equivalence ratios showed that rich mixtures occurred in some of the tests, indicating that some features of confinement

were found in the raw gas analysis. These rich mixtures produced high concentrations of toxic gases. The 3 samples had similar total peak HRR (180-200 kW/m<sup>2</sup>), but the time variation of HRR was different with the CFM having a slower

growth of the fire to peak HRR. The substrates covered by melamine or maple, organic improvers, wax, and resin appear to have a chemical flame retardation effect that delayed the initial combustion.



**Fig. 4:** Mass loss rate (a), Equivalence ratio (b) and Heat release rate (HRR) (c)

**Toxic Gas Concentration**

The most important toxic gases are compared in fig. 5. High concentrations of the gases were released during the ignition delay and when the HRR was at its peak, for all the test fires due to the rich mixture at the initial stage of the fire. OSB had Formaldehyde and acrolein concentration higher than the other samples by a factor of 4. The most important toxic species

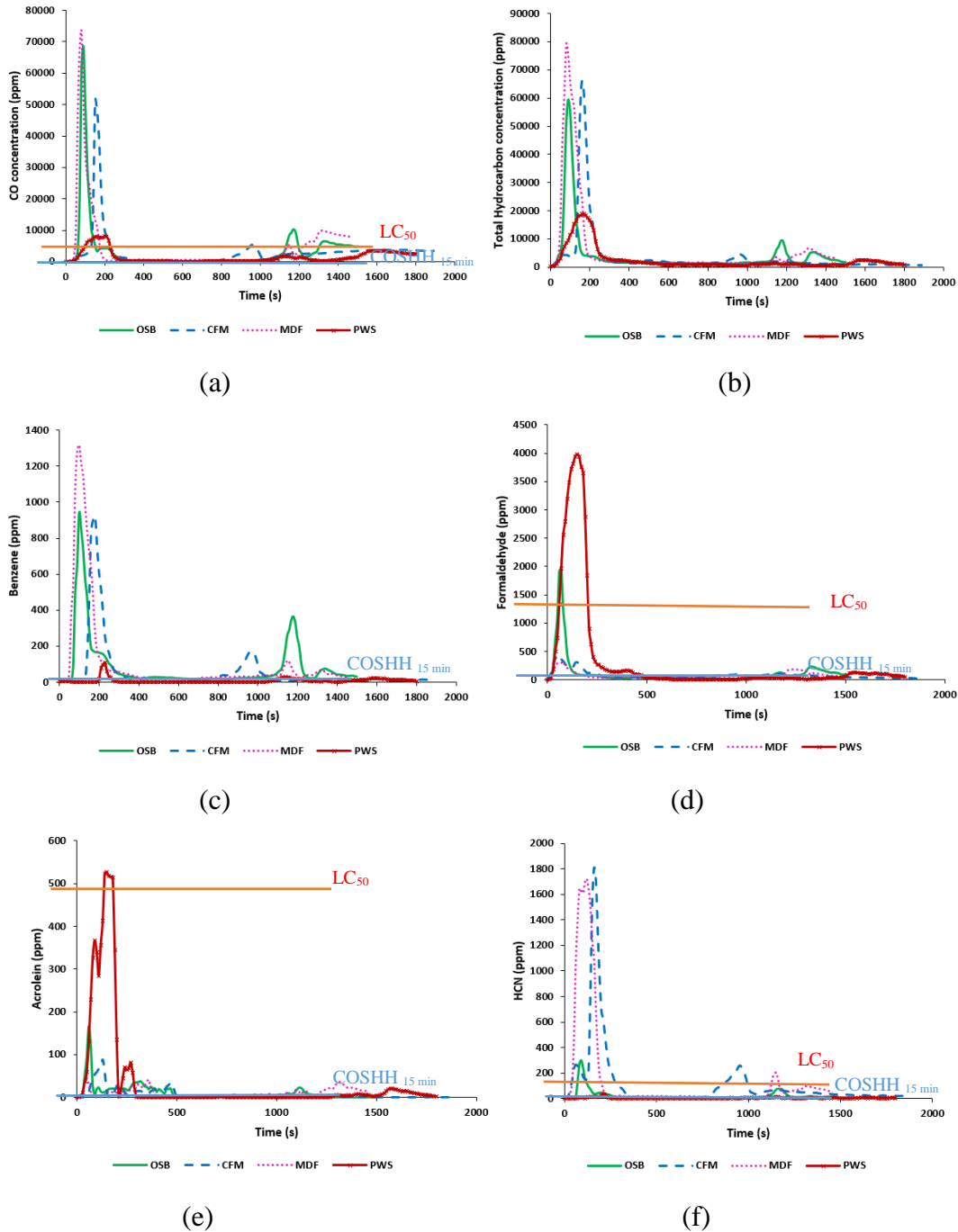
were CO, acrolein, formaldehyde, benzene and HCN on both an LC50 and COSHH<sub>15min</sub> basis, which is used as an indicator of impairment of escape.

HCN concentration was very low for OSB which was expected from the low nitrogen content in Table 1. The MFC and MDF had high organic nitrogen in Table 1 and high HCN in Fig. 5f. The high nitrogen content in the MFC



and MDF was due to their composite structure with glued chips, particles or fibres. All concentrations exceeded the toxic limits in terms

of  $LC_{50}$  and  $COSHH_{15\text{ min}}$  for all toxic species in Fig. 5, except for benzene  $LC_{50}$  which is a concentration limit of 10,000 ppm.

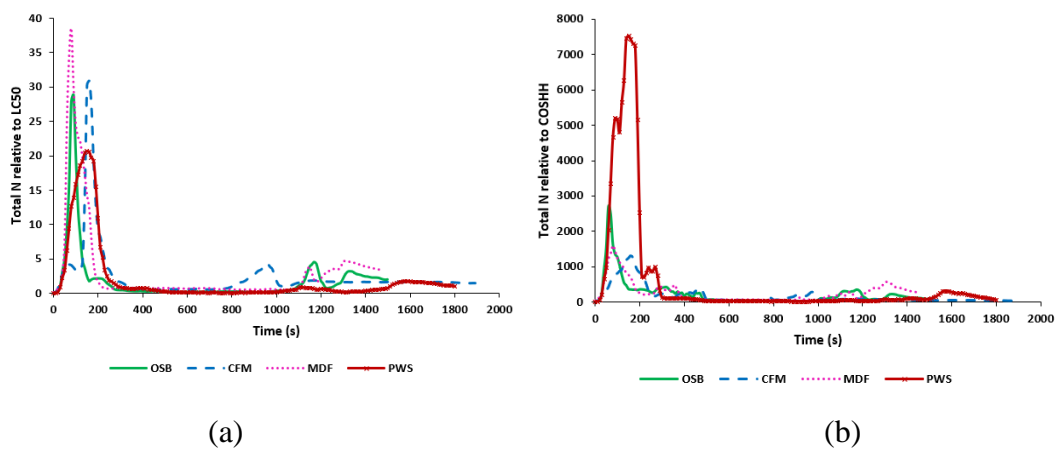


**Fig. 5:** Toxic gas concentrations; CO (a), Total Hydrocarbon (b), Benzene (c), Formaldehyde (d), Acrolein (e) and Hydrogen Cyanide (f).

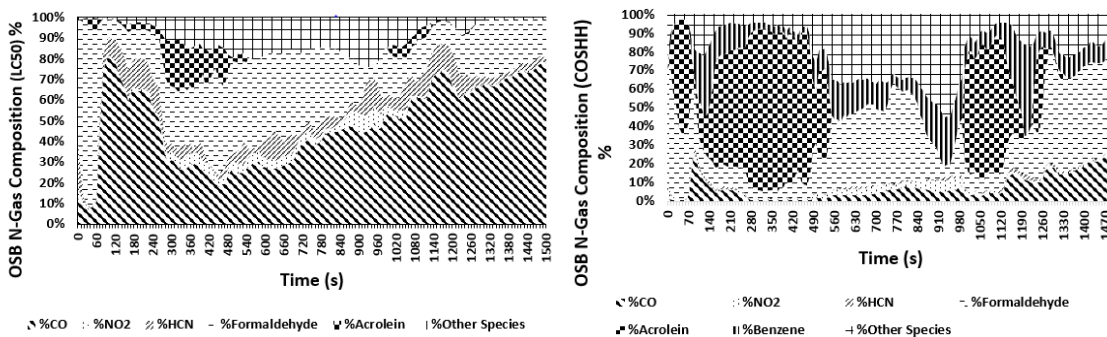
**Total Toxicity**

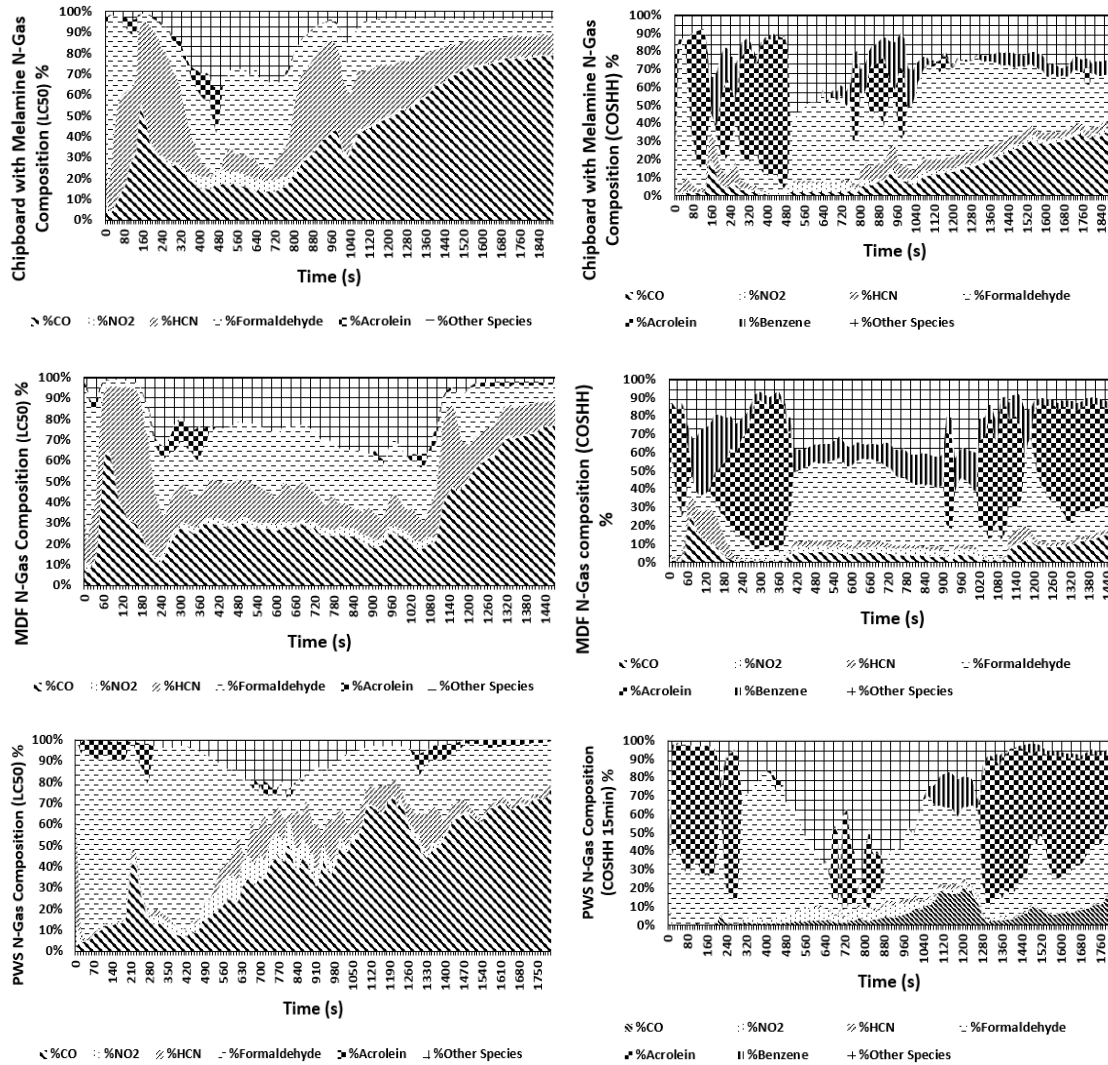
The total toxicity N for the 3 wood samples is shown in fig. 6 as a function of time. This shows a similar variation of N with time for the COSHH<sub>15min</sub> and LC<sub>50</sub> toxic assessments. At the early combustion stage (140-200 s), lethal levels of 30-minute exposure toxicity were produced in these wood fires and the COSHH<sub>15min</sub> toxicity levels indicate that impaired escape would occur for the entire duration of the fire, even though

the fire burnt lean for most of the time. The dilution required to prevent 30-minute LC<sub>50</sub> exposure levels from being lethal was about 30-40 indicating that people exposed to these gases would be at risk of death. Impairment of escape would be a much more significant effect as these toxic gases need to be diluted by over 1000 for the MFC fire and over 2000 for OSB and MDF before these gases would not impair escape.



**Fig. 6: Total toxicity N relative LC<sub>50</sub> (a) and relative to COSHH<sub>15 min</sub>**





**Fig. 7:** Toxic Composition of N-gas (LC<sub>50</sub> relative and COSHH<sub>15 min</sub> relative) as a function of time

Figure 7 shows the key toxic gases that would cause death and impair escape in the processed wood fires. On LC<sub>50</sub> basis, the key gases were found to be CO, NO<sub>2</sub>, HCN, Formaldehyde and acrolein while on COSHH<sub>15 min</sub> basis, the most important toxic gases were CO, NO<sub>2</sub>, HCN, formaldehyde, acrolein and benzene. This shows that for both toxicity assessment methods, most of the gases were common except for benzene which the LC<sub>50</sub> does not give much importance. CO is the most dominant toxic gas in terms of

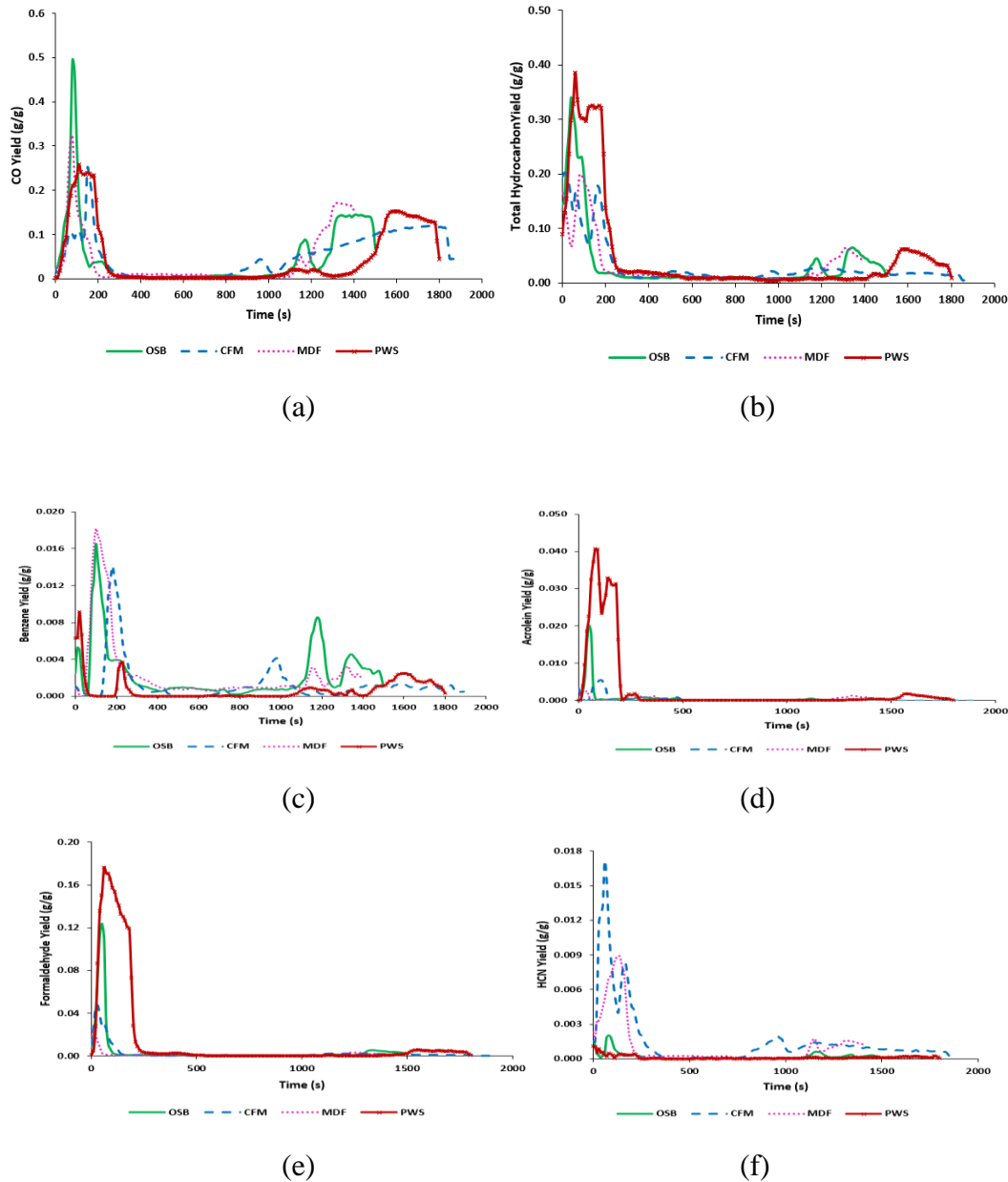
LC<sub>50</sub> while formaldehyde and acrolein are the most dominant in terms of COSHH<sub>15 min</sub>.

### Toxic Yields

The yields for the key toxic gases and their variations with time are shown in fig. 8. CO yield was higher in the initial stages with a reduced yield during the char burning phase as shown in fig. 8a. OSB had the highest CO yield with a peak of 0.5 g/g (500 g/kg) followed by MDF with a yield of 0.32 g/g (320 g/kg) and CFM and pine produced similar yield of 0.25 g/g (250 g/kg). The total unburnt hydrocarbon yields

are shown in fig. 8b. These were much higher in the initial volatile burning phase and lower in the char burning phase. The processed wood produced higher yield of benzene as compared to the pine wood and these were produced at the initial stage of the combustion and the char burning phase. High yields of acrolein and formaldehyde were produced by pine during its ignition delay of 192 s as compared to the processed woods but once flaming combustion started, and during the char burning phase,

yields became very low. The nitrogen in the binders gave rise to the production of HCN with the CFM having the highest yield of HCN of about 0.017 g/g (17 g/kg) followed by MDF with a yield of 0.009 g/g (9 g/kg) and OSB with a yield of 0.002 g/g (2 g/kg). Comparing the yield of HCN with pine wood shows that pine produced a very low yield of HCN as expected because of the low nitrogen content of pine wood.



**Fig. 8:** Toxic Gas Yields

## CONCLUSION

The raw gas sampling showed there was confinement at the initial stage of the fire, creating rich mixtures and this created high levels of toxic gases. Processed wood fires released very high concentrations of these gases, impairing escape and potentially causing death. Melamine's flame-retardant properties prolonged the ignition of the MFC, while the glue and resins used in manufacturing wood-based composites posed a toxicity hazard by releasing

high concentrations of HCN and other toxic gases. This research also showed that the modified standard cone calorimeter is a good technique to use for the realistic determination of toxic gases when used with the heated FTIR.

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