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Ignition and Toxicity Evaluation of Selected Aircraft Interior Materials Using the Cone Calorimeter and FTIR Analysis

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ABSTRACT

Aircraft fires are more critical than building fires as there is no immediate means of escape when in flight and the fire compartment is a closed space with fixed ventilation. The toxic gas yields were determined for aircraft interior material fires. Fabric seat covers and wall/floor covering were tested using a cone calorimeter with 40 kW/m² heat flux and an enclosed restricted ventilation compartment with 15 air changes per hour (ACH), which is typical of aircraft passenger compartment ventilation. The cone calorimeter was modified to enable raw gas samples to be taken from a chimney on the fire compartment exhaust. The fire effluents were analysed using a heated online Fourier Transform Infrared (FTIR) analyser which determined 64 species in the fire products. Different burning patterns were observed for each of the materials with the Fabric seat cover sustaining a flaming fire for 27 minutes while the wall/floor cover had only a 33s flaming period. The major toxic emissions for the fabric seat cover were SO_2, HCN and CO and for the wall/floor cover were HCN, CO and NH_3.

INTRODUCTION

Inhalation of toxic emissions released from fires is the major cause of death for casualties from fire incidents [1]. Released smoke in compartment fires reduces visibility and contains irritant gases, which causes impaired vision and respiratory problems [2]. Toxic emissions (asphyxiant and irritant) gases cause impairment of escape and result in an increase in the evacuation time and possible injury and/or death.. A fire in the passenger compartment of an aircraft is more hazardous than a building fire, as in flight, there is no escape and there is restricted ventilation of the fire with the fire products recirculated. Also ceiling heights are low so that smoke logging occurs much more quickly than in buildings. Thus, it is crucially important that aircraft passenger compartment materials are difficult to ignite, do not easily propagating fires and have minimum yields of toxic emissions.

Saudi Arabian Airline flight 163 is an illustrative example [3] of the large number of deaths that occur in aircraft passenger compartment fires. The pilot managed to land the aircraft but that was not enough to save anybody's life that day, because no one managed to open the airplane door to escape as everybody was incapacitated by toxic emissions, and the doors were closed until they were opened by the rescue services from outside. British Airtours 1985 disaster in Manchester [4] was a major incident in the history of aviation safety with a significant impact on the regulations especially in terms of recommending stricter limitations on interior cabin materials in terms of potential soot emissions, in case of involvement in a fire. In air starved fires such as in aircraft compartments, the potential risk of backdraught when opening the exit door for evacuation is always present. This was the case for Air Canada flight 797 [5] The fire propagated in the cavity ceiling generating unburned hydrocarbons resulting in a backdraught only 90 seconds after opening the door for evacuation.
Current fire tests for selecting material for aircraft interiors only look at the ease of ignition and the speed of flame propagation under freely ventilated conditions [6]. There are no standards to assess the toxicity once the material is engulfed in a fire [7] apart from those relating to visible smoke release. Usually the rate of smoke production is measured using optical obscuration, rather than the more useful filter paper based gravimetric methods [8]. This work was aimed at showing that the cone calorimeter could be modified to assess fire toxicity using samples of aircraft materials burnt with the same air ventilation as in aircraft passenger compartments, which is typically 15 ACH.

Andrews et al. [9] investigated compartment fires with restricted ventilation at 22 ACH, which was similar conditions to those in aircraft passenger compartments. The fire compartment was 1.56 m^3 and was instrumented with thermocouple arrays and the toxic gases were sampled at the ceiling mixed gas discharge point. They investigated a fire load that was mainly the acrylic blankets and pillows issued to all passengers, with some other materials issued per passenger. Fire temperatures were relatively low at maximum of 300^oC and the heat release peaked at 200kW. The restricted ventilation limited the development of the fire. The key toxic gases, using the same FTIR system as in the present work, were HCN which peaked at 4600ppm, SO_2 with a peak at 470ppm and HCl which peaked at 1% with the source of chlorine from fire retardants. There were also significant levels of CO and formaldehyde. The three most important toxic gases relative to their critical toxic levels were HCl, HCN and formaldehyde which accounted for 94% of the total toxic N. These are quite different results from equivalent studies using cotton [10] and pine crib fires [11] in the same ventilation controlled compartment. For these materials the key toxic gases were CO, formaldehyde and acrolein with smaller contributions from benzene and acetic acid. For HCl and HCN to be found in the toxic gases the fire load has to contain Cl and N in their elemental composition or in the fire retardants and this is a feature of the two aircraft materials studied in the present work. This work is part of a larger project investigating aircraft passenger compartment toxic gases and all the materials studied were part of the aircraft furniture and fittings.

EXPERIMENTAL EQUIPMENT

A CE Flash EA2000 elemental analyser was used to determine the HCONS analysis for the aircraft material samples. A LEO1530 Gemini Field Emission Gun Scanning Electron Microscopy (FEG-SEM) with Energy Dispersive X-ray Detection (EDX) analysis was used to determine the composition of wall/floor cover large ash content. The EDX analyser was able to detect all elements in the periodic table except H, He, Li, and Be (any element without electrons in the p shell i.e. elements with atomic number less than 5) [12]. A Mettler Toledo TGA/DSC 1 instrument was used to perform thermogravimetric analysis for water, volatiles, carbon and ash. Samples were run in nitrogen at 25°C, heating at 10°C/min to 100°C, and then held for five minutes to remove the moisture content. Then it was heated at the same rate to 700°C which was held again for another five minutes evaporating volatiles before purging air into the sample combust any carbon, with the remaining weight being ash.

A Gasmet FTIR CR-2000 instrument with 190^{o}C heated detection cell was used for toxic gas analysis. The FTIR had a 2 meter path length multi-pass sample cell with a volume of 0.22 l. The FTIR sample cell was coated with rhodium-gold non-porous protective layer resisting corrosion. To produce a time-averaged spectrum, a liquid nitrogen cooled MCT detector was used with10 spectra scans per second and scans were averaged over 10s. The longer the time average, the more improved was the signal to noise ratio and more species can be resolved in each analysis. The Leeds FTIR was calibrated for the 64 species likely to be encountered in combustion processes, including most of the toxic gases of interest in fire toxicity, over a range of reference concentrations for each.

The materials were tested for toxic gas release using small 100mm square specimens on a cone calorimeter, modified to enable ventilation controlled combustion and direct undiluted sampling of the products of combustion. The standard cone calorimeter dilution was still used for the determination of the heat release rate (HRR) by oxygen consumption. The cone calorimeter also determines the smoke generation by the obscuration method. The cone calorimeter has a truncated conical radiator with a range of heat fluxes 10-100 kW m^{-2} applied as a constant heat flux across the surface of the specimen with a thickness of (5-50 mm). The material to be tested in the 100mm square mount is mounted on a load cell 25mm below the cone. The fire products were collected by a hood and duct directly above the cone where O_2 concentrations and smoke density are measured [13, 14] . The apparatus original set-up is freely ventilated which makes it able to meet the ISO 19706 fire stages 1b for non-flaming tests and stage 2 for flaming tests [15].

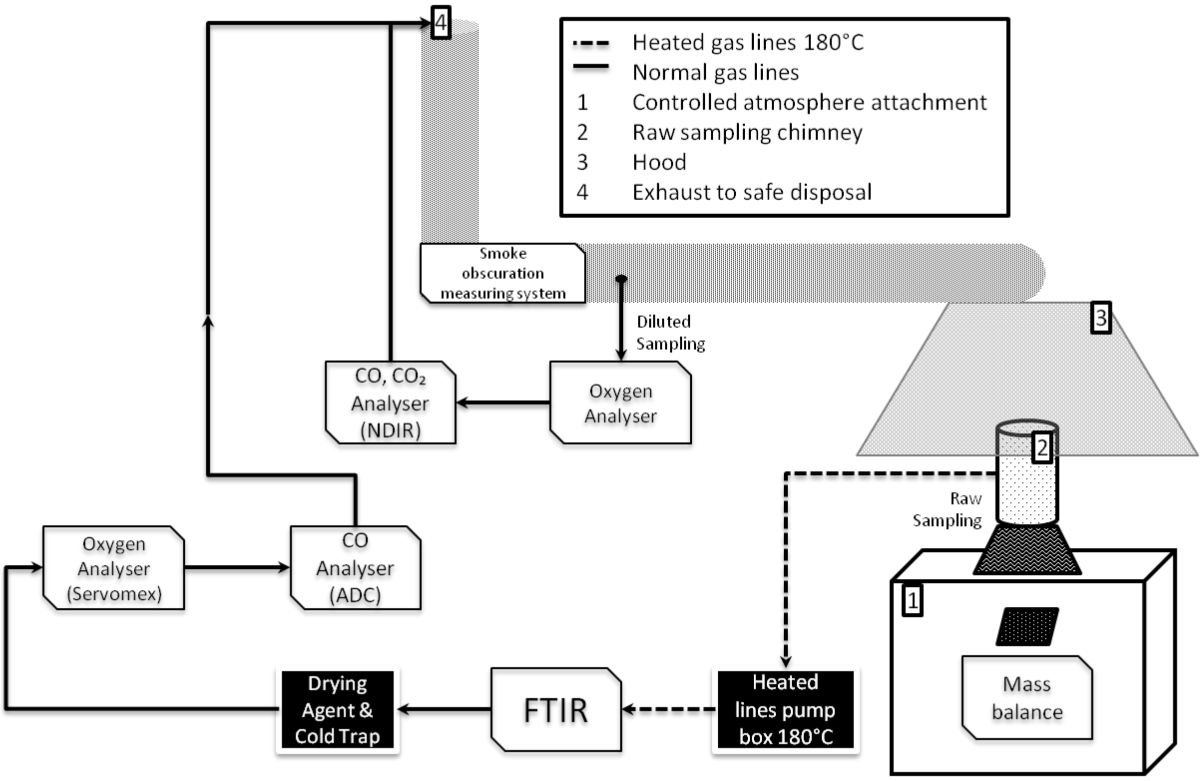

Figure 1: Diagram of the gas sampling system used on the cone calorimeter

The cone calorimeter had two modifications for this work (see Figure 1): firstly, it was modified to generate a compartment around the test specimen so that the air flow to the fire could be regulated and secondly, it was modified to enable raw fire gas product analysis without external air dilution to be sampled. Similar enclosures have been used by other researchers for testing materials in an inert or reduced oxygen environment. In this work metered air was supplied to the chamber to achieve the desired ACH. Raw combustion products from the ventilation controlled fire chamber were sampled by adding a 210mm long exhaust pipe on the conical heater central hole with the same internal diameter (80mm) as the cone upper opening. This acted as a chimney and enabled a gas sample probe to be inserted into this outlet ‘chimney’, without influencing the entrainment of air into the conical outlet. This procedure eliminating any post-oxidation before the sampling point, which is the criticism made of previous publications of fire toxicity measurements using the standard cone calorimeter [2, 16, 17]. This would still leave the fire gases in contact with the conical heating element, but this is considered to be a negligible effect as the temperatures are too low for significant reaction.

Oxygen consumption calorimeter was also used on the raw sample gas as well as the conventional diluted oxygen consumption HRR with the standard cone calorimeter system, which was unaffected by the addition of the cone discharge chimney. This enabled the proportion of the total heat release that was released in the ventilation controlled compartment to be determined. Essentially these modifications of the cone calorimeter convert it into an equivalent apparatus to flame propagation apparatus [18] where a fire sample on load cell is placed in a vertical quartz tube with an external conical heater and metered airflow up the quartz tube. FPA been used heavily by Tewarson in producing his gases yield database [19]. These modifications enable the apparatus to meet ISO 19706 fire stages 3a & 3b [15] for under ventilated flaming tests.

The modified cone calorimeter was used with 15ACH restricted ventilation. This simulates the ventilation conditions in the aircraft cabin in accordance with a survey conducted in 1994 on commercial airlines [17]. The heat flux was set at 40kW/m² which represented a surrounding flaming fire, as suggested by the classification of fires in the ISO19706 [15]. The raw gas analysis from the modified cone calorimeter chimney was sampled using a single point stainless steel tube into a 190°C heated sample line. This heated sample line was connected to a heated pump and sample filter system and then connected to the FTIR through another heated sample line. Finally the FTIR detection chamber was also fully heated at 190°C. This ensured no loss of toxic species due to condensation or solution in water. SO_2, HCl, NO_2 and many other species such as aldehydes are all lost or reduced in concentration if they contact condensed water in the sampling system.

The importance of the heated sampling lines, filter and heated exhaust in the analysis of toxic gases from fires using raw gas samples is shown in Table 1. This shows the losses of toxic gases that occur if water condenses in the sample lines, sample pump or sample filter. In the present work it will be shown that the key toxic gases were HCl, HCN, SO_2 and formaldehyde and all would be lost in solution in water if the sampling and analytical system was not heated. This sampling problem from raw fire gases is at the heart of the use of dilution of fire products prior to analysis. Dilution lowers the dewpoint of the sample so that the water vapour in the products of combustion does not condense when the sample is cooled by dilution. The problem of this approach is that the concentration is reduced to a level that makes analysis difficult. The dilution of the raw gas sample in the present work is about a factor of 300 and this would reduce raw gas concentrations from the 100s of ppm into the <1ppm range, where the current FTIR is not sufficiently sensitive and mass spectrometry with ppb resolution would be required. This is a key reason for the lack of data on toxic gas yields from fires other than for CO.

Table 1: The problem of sample losses in unheated raw gas sampling and analysis systems

Species	Boiling Point	Solubility in Water at 20°C	Heated Sampling System?
CO	-192°C	0.006 g/litre	No
Benzene	80°C	0.8 g/litre	Yes >80°C
Naphthalene	218°C	0.03 g/litre	Yes > 220 °C
Formaldehyde	96°C	Extremely Soluble	Yes > 100°C
Acrolein	53°C	Very Soluble	Yes > 100°C
NO_2	21°C	Hydrolyses to HNO_3	Yes > 100°C
HCN	25.6°C	Completely Miscible	Yes > 100°C
HCl	-85°C	Extremely soluble 720g/litre	Yes > 100°C
SO_2	-10°C	Extremely soluble 80 vol/vol	Yes > 100°C

RESULTS AND ANALYSIS

Aircraft Passenger Compartment Samples used in the Fire Tests:

1. Fabric Seat Cover: the elemental composition was 48.2% C, 6.2% H, 26.1% O, 15.0% N, and 4.5% S giving a stoichiometric air to fuel ratio on a dry ash free basis (daf) of 6.47. The N content indicates that this was an acrylic type material and toxic HCN would be expected and was found in the outlet gases. The relatively high sulphur content, due to the presence of S content in flame retardants, would result in SO_2 emissions and these were found to be a major factor in the overall toxicity. The calorific value based on the elemental analysis was 24.1 MJ/kg. The TGA analysis is shown in Table 2. The high volatile content resulted in flaming combustion. A 300×300 mm cut of the fabric was folded to fit the sample holder and to have a thickness of 7 mm and initial mass of 44.6 g.

2. Wall/floor (W/f) cover: the elemental composition was 33.4% C, 5.2% H, 45.6% O, 9.1% N, 3.4% S and 2.5% Al with a daf stoichiometric air to fuel ratio of 3.39. The calorific value was 18.2 MJ/kg. The N and S content would also lead to HCN and SO_2 in the toxic gases. The presence of Al indicates that AlOH fire retardant was used in this material. The calorific value based on the elemental analysis was 18.2 MJ/kg. The TGA analysis is shown in Table 2. The high ash content indicates the present of mineral based fire retardants such as carbonates and the CO_2 results confirmed this. Six 100×100 mm pieces were cut and put over each other to form a thickness of 6.6 mm and initial mass of 46.2 g.

Table 2: Proximate analysis of the samples before and after the test

	Fabric seat	Fabric seat	W/f cover	W/f cover

	cover (Raw) [wt%]	cover (Debris) [wt%]	(Raw) [wt%]	(Debris) [wt%]
Water vapour	4.7	4.6	3.7	4.8
Volatiles	68.3	2.5	48.8	3.7
Fixed Carbon	25.0	62.4	30.1	19.9
Ash	2.0	30.5	17.4	71.5

Fire Development

The mass loss of the samples in the fire is shown in Figure 2. The fabric seat cover had a significant flaming stage for 27min losing 75% of the original mass before the non-flaming smouldering stage started, which continued for 22min losing an additional 7% of mass by the end of the test. The wall/floor cover sample had a brief flaming stage for only 33sec and lost less than 4% of its initial weight before the specimen entered the non-flaming smouldering stage where 74% of the original weight was lost in 39min. The mass loss rate was stable in the range of 0.9-1.2 g/min until the 1500th second by which time half of the material was lost, then the mass loss rate dropped to lower values. These results indicate that the 49% volatiles with this material was not hydrocarbons and hence did not give rise to extensive flaming combustion. It is considered that the volatiles measured in the TGA analysis was from the decomposition of AlOH and carbonates to give H_2O and CO_2 respectively. The heat absorbed in this decomposition and the release of CO_2 was the mechanism of the fire retardancy which resulted in non-flaming smouldering combustion.

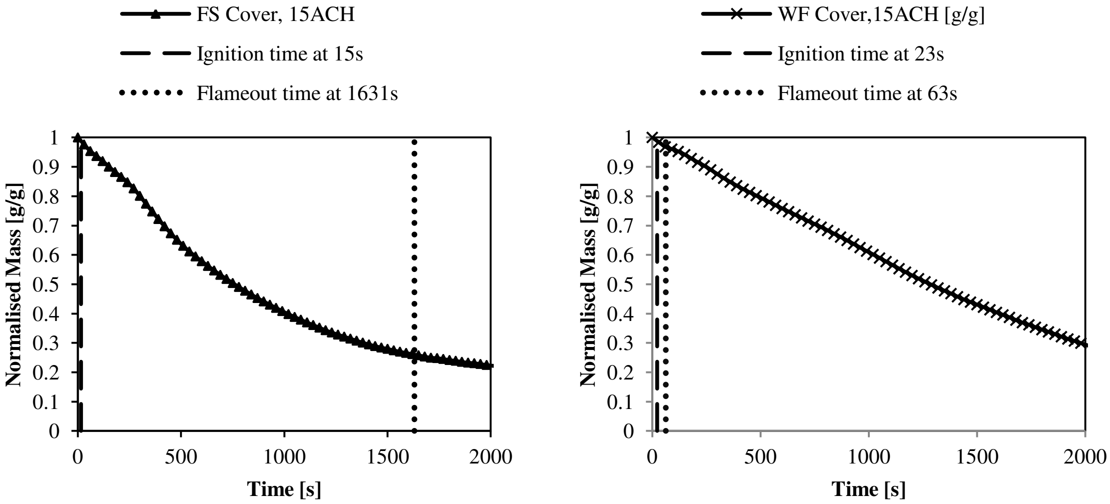

Figure 2: Normalised mass versus time, indicating flaming and non-flaming stage (Left: fabric seat cover, Right: wall/floor cover)

Heat Release Rates

Figure 3 compares the three methods of evaluating the heat release rate (HRR). The three methods are:

1. HRR based on the mass loss in Figure 2 multiplied by the material calorific value.
2. HRR based on the oxygen consumption from the raw chimney gas analysis.
3. HRR based on the conventional cone calorimeter total HRR including post chimney oxidation

Figure 3 shows distinct differences between the three HRR. The raw gas fire HRR was the lowest HRR and was significantly below that based on the cone calorimeter HRR. This determines the HRR due to post compartment oxidation as air is entrained into the hot gases emerging from the chimney. This difference is particularly large for the wall/floor covering, where there is about 15 kW of post primary combustion. These results show that at 15 ACH these materials would not burn to completion due to lack of oxygen in the compartment. The consequence is a very high production of partially burned toxic gases, as discussed in the next section. These results also show that evaluation of toxic gases under freely ventilated fire conditions, as in the standard cone calorimeter, will give false results and unrealistically low values of toxic gas yields. These results also show that if post primary combustion air dilution is used prior to toxic gas analysis, as in the Purser furnace method [1, 2], then toxic gas yields will also be underestimated due to oxidation in the dilution process. The Purser furnace method uses less dilution air than in the cone calorimeter method, but the dilution factor can be as high as 25 when rich fire combustion is studied, which is the condition for greatest post furnace oxidation.

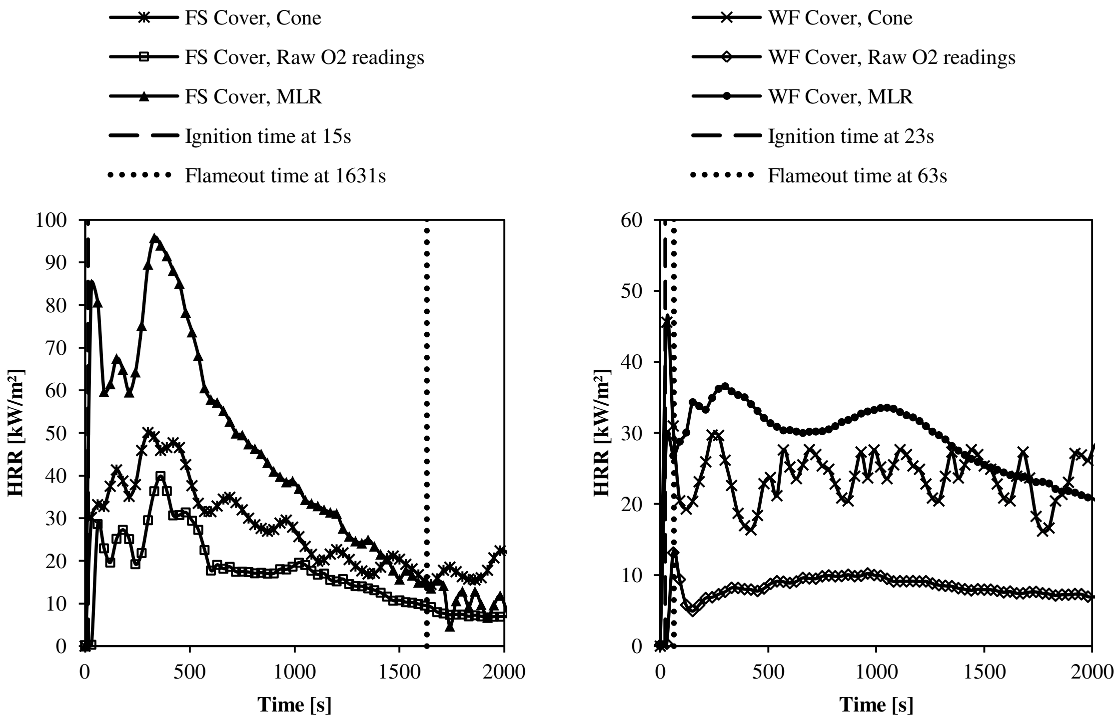

Figure 3: HRR versus time, flaming and non-flaming stages of fire shown for (Left: fabric seat cover, Right: wall/floor cover)

Figure 3 also shows that the HRR based on the mass loss time the calorific value results HRR values that are too high as they are much higher than those determined by the cone calorimeter. The reason for this is that there are other gases released in the tests that lead to mass loss, but not through combustion. This is the decomposition of fire retardant AlOH in the wall/floor material. Another source of fire retardant weight loss that could be significant is that of the decomposition of carbonates in the base material to yield CO_2.

Figure 3 shows that the fabric seat cover's flaming stage reached a peak HRR 50 kW/m^2 before it reduced to less than 20 kW/m^2 after flameout in the smouldering stage. The wall/floor cover had a peak HRR in the brief flaming stage of 50kW/m^2 then the smouldering stage combustion had a stable HRR of around 25 kW/m^2 for the rest of the test. These results demonstrate the efficient flame retardancy in the wall/floor covering, but a lack of effective flame retardancy in the fabric seat cover where there was an extensive period of flaming combustion that lasted 1600s. However, the heat release in the smouldering combustion phase of the wall/floor covering was only about 10kW and the yield of toxic gases was significant, which were oxidised in the post chimney dilution process.

Toxicity Results

The toxicity produced from the fires was evaluated using a modified N-gas model [20]. The N-gas was obtained by dividing the concentration of the species by the LC_{50} values and these ratios were summed-up to produce the total toxic gas N ratio. This is a measure of the risk of deaths in fires. A similar analysis done based on $COSHH_{15min}$ or $AEGL_{10\ min}$ toxicity data, which relates to impairment of escape. The LC_{50} limit represents the lethal concentration which kills half the test animals group [1] after 30 min exposure, and within a specified period of time of post exposure monitoring. In contrast the COSHH 15 and AEGL 10 minute represent a maximum safe exposure limit (for the period stated) or the safe means of escape with no significant toxic gas exposure.

Table 3: Comparison of the LC_{50} and $COSHH_{15min}$ toxic gas limits

Gas	**formula**	**LC_{50} [ppm]**	**$COSHH_{15min}$ [ppm]**	**LC_{50}/COSHH**	**ratio to CO**
Carbon Monoxide	CO	5,700	200	28.5	1.00
Hydrogen cyanide	HCN	165	10	16.5	0.58
Ammonia	NH_3	750	35	21.43	0.75
Sulphur dioxide	SO_2	1,400	5	280	9.82
Formaldehyde	CH_2O	750	2	375	13.16
Acrolein	C_3H_5O	150	0.3	500	17.54
Hydrogen fluoride	HF	2,900	3	966.67	33.92
Hydrogen Bromide	HBr	3,800	3	1266.67	44.44

Table 3 compares the LC_{50} and $COSHH_{15\ min}$ toxic limits for 8 toxic species. The ratio of the two limits $LC_{50}/COSHH_{15min}$ is also shown in Table 3 and this is clearly not the same value. Thus the two toxicity assessment systems are not giving the same relative importance to the different species. If they were the same relative toxic assessment then the ratio would be constant. Another way of illustrating this difference is to normalise the ratio to 1 for CO. For species where the normalised ratio is <1 the LC_{50} assessment rates that species more toxic relative to CO than does $COSHH_{15min}$. An example of this is HCN. Where the normalised toxicity ratio if >1 then the $COSHH_{15min}$ rates that species more toxic relative to CO than does LC_{50}. Acrolein is a key example of an important toxic gas that the two assessment methods disagree strongly on. It is thus clear that species such as acrolein will delay your escape through the action of toxic acidic gases but they will not kill you. Both methods of toxic gas assessment are used in this work.

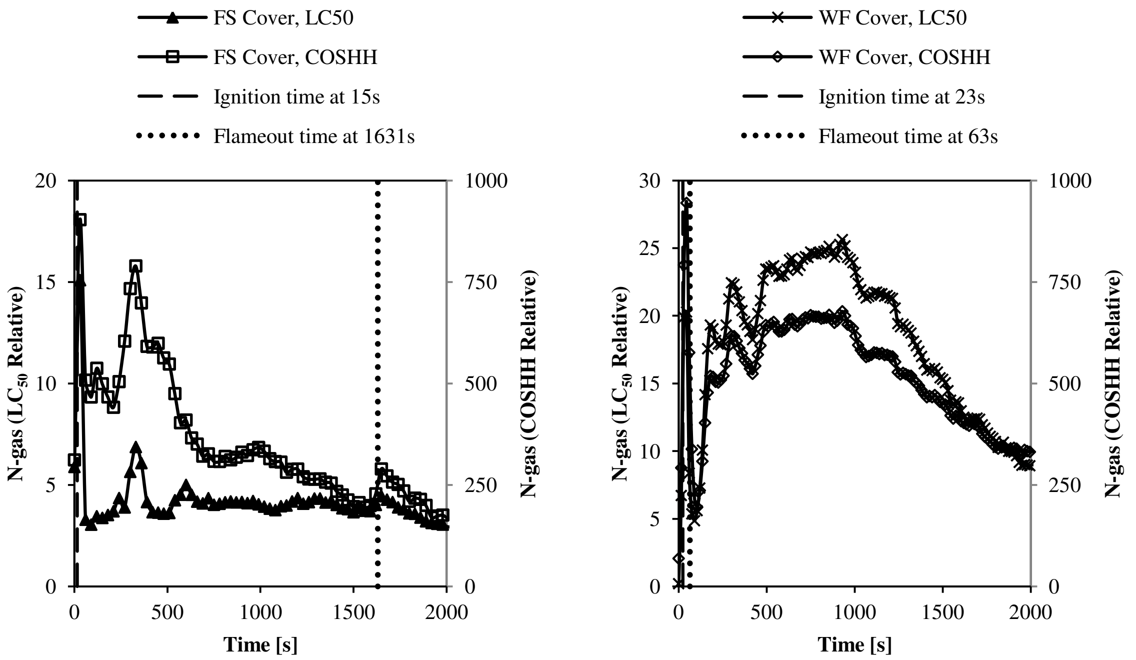

Figure 4: N-gas (LC50 relative) versus time, flaming and non-flaming stages of fire shown for (Left: fabric seat cover, Right: wall/floor cover)

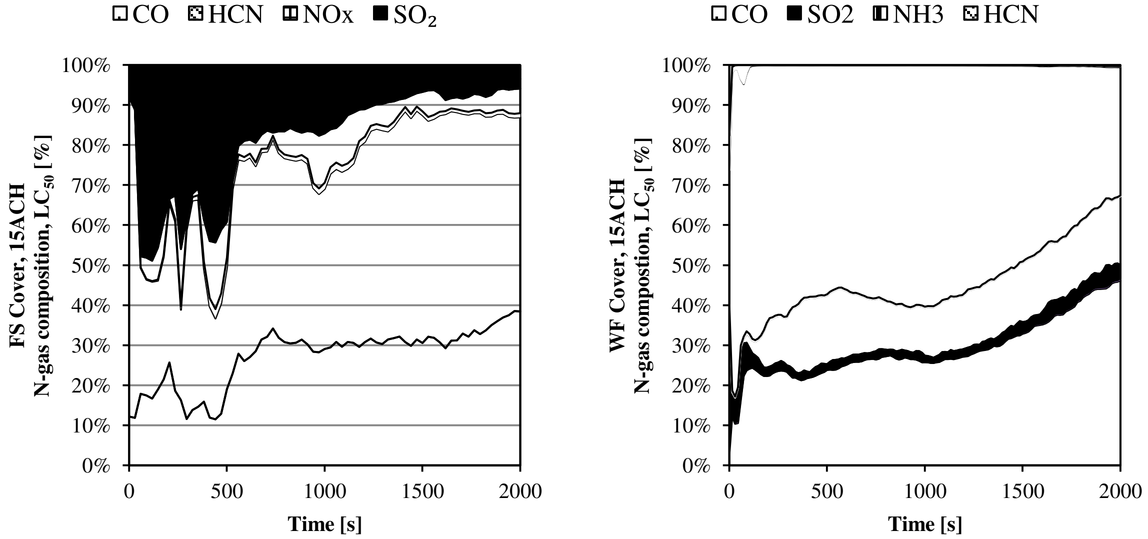

Figure 5: Toxic composition of N-gas (LC$_{50}$ relative) versus time shown for (Left: fabric seat cover, Right: wall/floor cover)

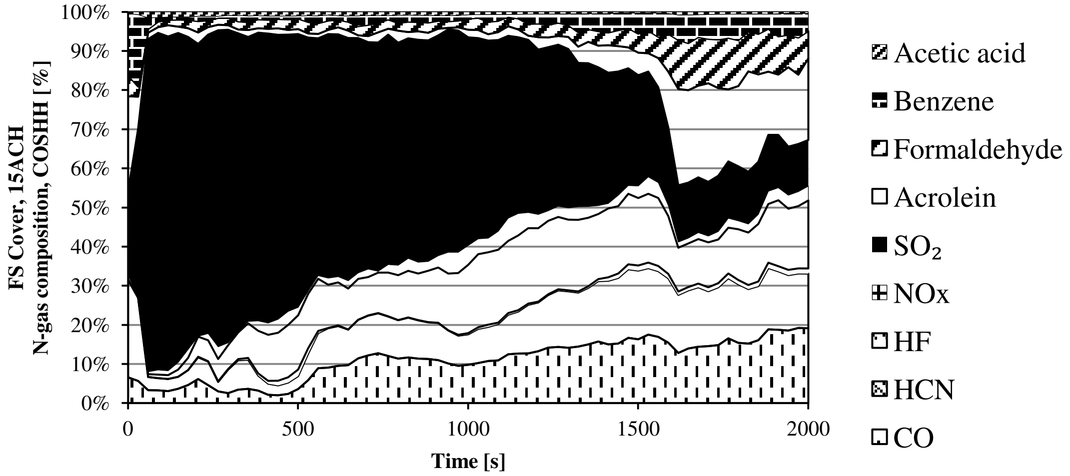

Figure 6: Toxic compostion of N-gas ($COSHH_{15min}$) for the fabric seat cover

Figure 5 shows that for risk of death in a fire involving these materials in an aircraft compartment, the key toxic gases are HCN, CO, SO_2 and NO_2.for the fabric seat cover. HCN was derived from the 15% N in the fabric seat cover material, which was most likely acrylic. Figure 6 shows that on a COSHH basis the most important toxic gases were more complex. The toxic gases that would impair the means of escape were in order of importance: SO_2, HF, CO, HCN. This shows that three of the toxic gases were common between the two assessment methods. The importance of HF was due to the use of a fluorine based fire retardant in the fabric seat material. In the work of Andrews et al. [9] for mainly acrylic blankets under aircraft passenger compartment ventilation the most important toxic gases were HCl, HCN and formaldehyde. The lack of HCl in the present results indicates that no chlorine based fire retardants were used in the two materials tested.

Figure 5 also shows for the wall/floor covering material that the most important LC_{50} toxic gases were in order of importance: HCN, CO, NH_3 and SO_2. The 9.1% N content of the material was the source of HCN and NH_3 through local rich burning. The 3.4% S was obviously the source of the SO_2. Figure 6 shows that on a COSHH basis the most important toxic gases were more complex. The toxic gases that would impair the means of escape were in order of importance: HCN, CO, SO_2, NH_3, Benzene, formaldehyde and NO_2. It is clear that the toxic gases in fires are very dependent on the materials used. Some toxic gases could be eliminated if materials were screened for undesirable elements that would give rise to dangerous toxic gases. Elimination of N, S, F and Cl would be beneficial, but this would eliminate halogen based fire retardants and do this is not realistic. However, eliminating acrylic materials would be possible.

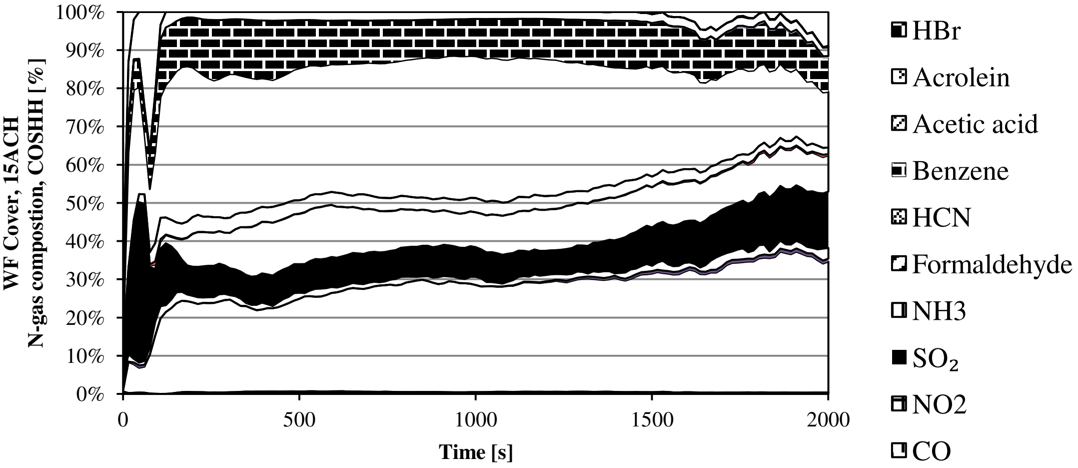

Figure 7: Toxic compostion of N-gas ($COSHH_{15min}$) for the wall/floor covering

Toxic Gas Yields, g/g.

The toxic gas yields were computed from the FTIR concentration and the A/F of the raw gas analysis based on carbon balance using Eq. 1

$$\textbf{Yield} = \textbf{K C } (1 + \textbf{A/F}) \qquad \textbf{[1]}$$

Where K is the conversion coefficient, which is the ratio of molecular weight of a specific pollutant to the molecular weight of the exhaust gas. The molecular weight of the exhaust gas is close to that of air (29). K is a constant for a particular toxic gas e.g.0.969 for CO.

C is the concentration of the component. If this is measured in ppm or % then the equation has to be multiplied by10^{-6} or 10^{-2} respectively.

A/F is the air/fuel ratio on a mass basis measured by carbon balance from the FTIR analysis.

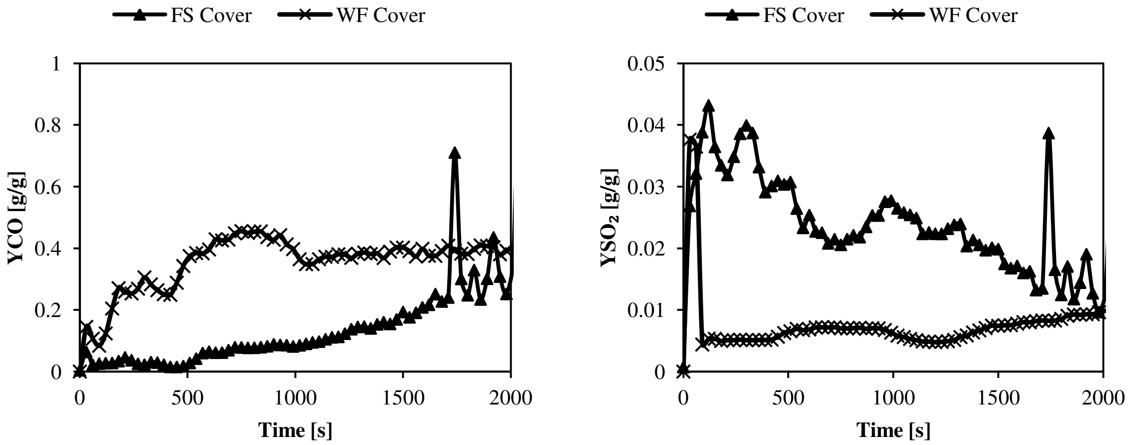

Figure 8: Toxic gas yields for CO (left) and SO_2 (right) for fabric seat covers and wall/floor covers.

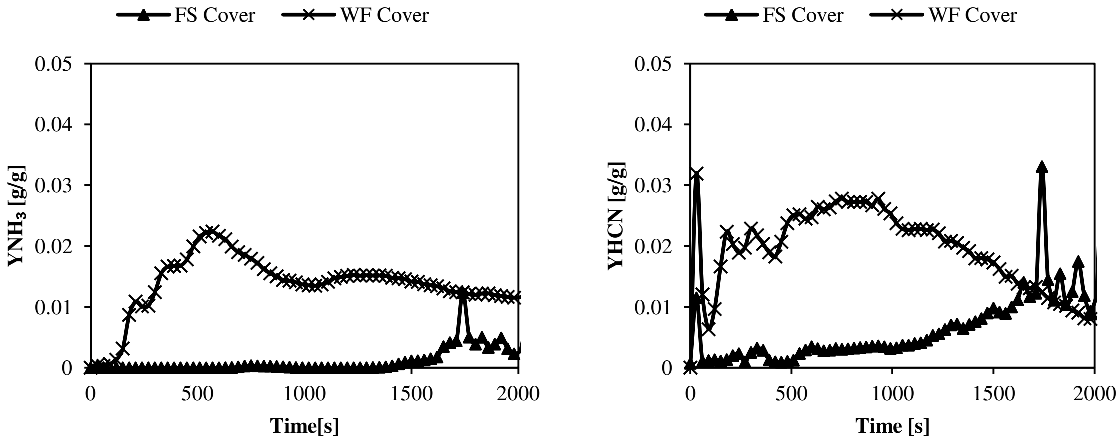

Figure 9: Toxic gas yields for NH_3 (left) and HCN (right) for fabric seat covers and wall/floor covers.

Figure 8 shows that the CO yield was much higher for the wall/floor covering than the seat fabric and this was also the case of NH_3 and HCN. However, for SO_2 the yield was much higher for the seat fabric. This indicates that the smouldering combustion of the wall/floor covering led to a much higher toxic yield than for flaming combustion of the seat fabric, apart from the yield of SO_2 where the flame led to much higher conversion to SO_2 with the seat fabric. The elemental analysis can be used together with the toxic yields to show that for the fabric seat cover 31% of the sulphur was converted into SO_2 compared with 11% for the wall/floor covers. For nitrogen mass balance the wall/floor cover with smouldering combustion converted 13% N to NH_3 and 18% to HCN. In contrast the flaming combustion of the seat fabric converted only 0.17% of the N to NH_3 and 6.5% to HCN. Most of the remaining N would be emitted as NOx with some converted to N_2 in rich burning regions close to the material surface.

CONCLUSIONS

- The FTIR analysis showed that Sulphur Dioxide was a major toxic gas for the aircraft fabric seat covers and a minor one for wall/floor covers. The sulphur most likely came from the fire retardant.
- The flaming burning for wall/floor cover was clearly suppressed much earlier than the fabric seat cover which demonstrates a greater flame spread resistance. However, the results also clearly show that persisting heat flux from other burning materials (represented by the cone heater in these experiments), will result in sustained smouldering contributing significantly to the toxic gases yields. This is not currently recognised in the screening of materials for use in aircraft interiors.
- HCN was the second most important toxic gas and this was due to the use of acrylic fibres in

the seat and wall coverings, which could easily be substituted for non-N-containing materials.

- Carbon monoxide was not the dominant toxic species, even though the materials were burnt under air starved conditions where CO is often the dominant toxic effluent.
- Toxic gases from these aircraft materials were different from normal building materials such as wood, where the major species are carbon monoxide, acrolein and formaldehyde.
- Raw gas sampling from compartment fires is the only way that the problem of post flame oxidation by dilution gases can be avoided and current toxic gas tests all involve post flame air dilution and hence underestimate the toxic yields.
- The cone calorimeter has been successfully modified to enable good toxic gas yields to be determined and should be considered as a reliable method for determining toxic gas yields in simulated compartment fire conditions with an imposed ventilation rate.

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