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Characterisation of Sparklers and Bengal Flames using the Fire Propagation Apparatus

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Abstract

Understanding the decomposition mechanism of fireworks is essential to improve the safety related to their storage (CHAF project). A study has been carried out on sparklers and Bengal flames to identify the nature of the combustion gases produced and investigate the potency of toxic emissions. Tests have been realised using the Fire Propagation Apparatus (FPA). It has become a widely used device for materials characterisation in fire research. It allows to directly measure the CO₂, CO, NO_x and THC productions. The emission of soot, the concentrations of HCl, SO₂ and HCN can also be obtained. From the experiments, it appears that the major gases produced were CO₂, CO and SO₂.

Pyrotechnics also present the peculiarity to carry an oxidiser and do not require O₂ from the air to burn. To explore this specificity, combustion experiments have been carried out in air as well as in nitrogen atmosphere. Results have been compared to underline the variations in the production of combustion gases depending on the reacting atmosphere. Under N₂ atmosphere, it emerges that the emission of CO₂ decreases while the CO increases.

In the future, other tests will be realised coupling FPA and FTIR to better characterise the emission of toxics.

Introduction

The two major stages where the risk related to the production and the use of fireworks become critical appear during storage and transport [1]. In the last few years, an increase in serious accidents involving fireworks has been observed. They often led to casualties and major damage to a large area where the event took place. One of the reasons suggested to explain this growth is that fireworks transport marking appeared to be inappropriate [1]. Several tests applied for material classification did not allow identifying a potential explosion hazard associated with the storage of fireworks in large quantities. The Quantification and Control of the Hazards Associated with the Transport and Bulk Storage of Fireworks project (CHAF project) was a recent initiative to develop a better understanding of fireworks behaviour in fires when large quantities are involved. The aim was then to provide methods allowing to identify fireworks likely to pose a mass explosion risk which would not be identified by current UN tests. Encouraged by results obtained, a larger consortium of laboratories, including INERIS, has decided to go further (CHAF2 Project), to suppress still remaining gaps in appropriate knowledge on those fireworks that may also lead to soil contamination and present acute or chronic toxicity issues at the work place level or the display stage for the workers [2]. Thus

learning more on intermediate or final decomposition products is deemed essential for progressing towards more sustainable fireworks industry. In order to be able to quantify the propagation of a reaction involving fireworks, the dominant parameters driving the phenomenon have to be recognized. In this perspective, understanding the decomposition mechanism of fireworks is essential to improve the safety related to their storage [3]. This characterisation requires adequate diagnostic instrumentation.

This work will investigate the ability of the FM-Global Fire Propagation Apparatus (FPA) [4] to provide appropriate data to determine the reaction process involved in the burning of fireworks. The FPA is a small scale calorimeter which became a widely used device in the field of fire research. It has proven its efficiency to characterise flammability parameters, related to the propensity of a material to support fire propagation [4]. Thermal and chemical characteristics the FPA is qualified to measure or estimate are time to ignition, mass loss rate, chemical, convective and effective heat release rate, combustion gases generation rates (CO₂, CO, Total Hydrocarbons (THC), soot...), O₂ consumption, the transient response to prescribed heat flux in a specified inert or oxidizing environment.

In the present work, an experimental study has been carried out on the ignition and the combustion of sparklers and Bengal flames to identify the nature of the major combustion gases produced and investigate the potency of toxic emissions. Atmospheres of the reaction area have been modified. In order to investigate the peculiarity of pyrotechnics mixtures to incorporate a fuel and an oxidizer in the same material, experiments have been performed under air and N₂ atmosphere. It will be of importance to analyse the influence of the O₂ concentration on the fireworks behaviour.

Experimental Approach

The experimental device used to perform the different tests is the FM-Global Fire Propagation Apparatus (FPA). It is a fire calorimeter (*cf.* Figure 1) which has been designed by Tewarson [5] in his early configuration in the middle of the 70's. During the last thirty years, the instrumentation received valuable improvement. Because it embodied several novel design concepts, it has been internationally recognised by standard organisation [4].

It comprises two main sections: the combustion line and the exhaust products (combustion gases, soot...) collecting and measurement line. The material submitted to a test is placed on a sample holder mounted on a load cell enclosed in a quartz tube delimiting the combustion area. Four infrared heaters fixed at the exterior of the quartz tube allows for applying a given external heat flux onto the sample. The heating system is both air and water cooled due to the high temperatures developed by the lamps. When self ignition is not expected, ignition of the material usually occurs by use of an ethylene/air pilot flame. When tests have to be performed under low O₂ or N₂ atmospheres, the usual ignition procedure could not maintain a flame. The ignition source is usually replaced by a sparks system. Air, O₂ and N₂ concentrations are introduced at the base of the apparatus (i.e. from the bottom of the sample holder). A hood system topped the "reaction zone". It has been dimensioned so that all the combustion gases are sucked up. From the hood, the system narrows to an exhaust duct to ensure gases mixing. Further down, volumetric flow rate is measured by a Pitot tube and gas sampling is operated. Gases pass through a soot filter and a cold trap. They are continuously distributed to a set of different analysers enabling to measure combustion gases concentrations. The O₂ concentration is estimated through a paramagnetic analyser while CO/CO₂ concentrations are determined using an infrared technology. A Flame Ionization Detector (FID) device is used to estimate THC level. A laser beam at $\lambda = 632 \text{ nm}$ is projected along the exhaust duct in order to evaluate the extinction and the

smoke molar fraction. Additional measurement procedures have been implemented to obtain the concentrations of HCl, SO₂ and HCN.

This device is adapted to the calorimetric methodology. It allows estimating the heat release rate (HRR) using oxygen consumption (OC) or carbon dioxide generation (CDG) calorimetry [6]. Data are acquired every two seconds for the duration of the experiments.

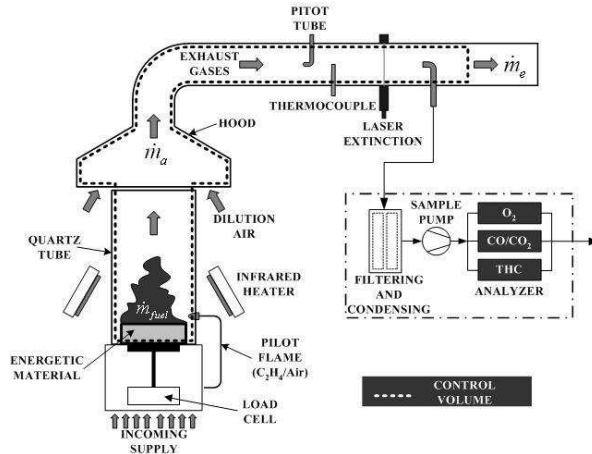


Figure 1. Scheme of the FM-Global Fire Propagation Apparatus.

Two types of fireworks have been tested, sparklers and Bengal flames. They are relatively common pyrotechnic materials. The compositions of the different samples are presented on Table 1.

Table 1. Compositions of the different tested fireworks.

Material	Colour	Composition		
Sparkler	White	100% KNO ₃	21% Sulphur	
		21% Charcoal	14% Aluminium	
Bengal Flames	White	100% KNO ₃	35% Sulphur	25% M ₁ *
		12.5% Antimony Sulphur	6.3% Aluminium	
	Yellow	100% KClO ₃	18.8% Sodium Oxalate	15.6% Shellac
		3.1% Acroides Gums		
	Red	100% KClO ₃	25% Strontium Carbonate	15% Resin
5% Acroides Gums				
	Blue	100% KClO ₃	40% Copper Oxychloride	8.7% Resin
*M ₁ :		100% KNO ₃	22% Charcoal	16% Sulphur

In order to obtain long combustion times and verify the homogeneity of the compounds, experiments were performed by coupling several samples together. For each test of sparklers and Bengal flames, respectively, 10 and 3 specimens were ignited. Samples were positioned in a way that they would burn one after the other allowing a reaction time long enough to be captured by the different measurement instrumentations. The Bengal flames were prepared in tubes with a diameter of about 5 mm. They were ignited by the top using a pilot flame or sparks.

Experimental Results

CO₂, CO, THC (Total Hydrocarbons), NO and HCl generation rates have been estimated from measured molar fraction. Mass loss was also recorded directly. Heat release rates were obtained from OC and CDG calorimetry. For the red Bengal flames, the CO₂ yield is significantly decreased by more than half while the CO generation substantially increases. On the other hand, combustion time decreases under N₂ atmosphere. The blue Bengal flame sees a slight decline of the CO₂ yield, combined with a low rise of the CO generation. As the blue Bengal flame seems to contain less fuel, the change from air to N₂ atmosphere in the reaction zone causes minor variation in the combustion gases concentrations. The yellow Bengal flame would represent an intermediate behaviour between the one observed with the red and blue compounds. The productions of THC, NO and soot do not seem to be affected when reaction takes place into a N₂ atmosphere rather than in air. No significant differences are measured regarding the combustion times.

Table 2 and Table 3 present mass losses, combustion times and generation yields obtained for combustion under air and N₂ atmosphere. Considering the combustion of sparklers, the percentage of mass burnt is relatively similar whether the reactive atmosphere was air or N₂. However, the combustion time is increased when the reacting atmosphere is changed from air to N₂. Generation rates diverge from one test conditions to another. CO₂ yield is decreased by more than half while the CO yield is multiplied by four when the atmosphere within the tube is changed from air to N₂. Higher level of THC is also observed.

Blue, yellow and red tested Bengal flames contained the same oxidizer, potassium chlorate (KClO₃). White contained potassium nitrate (KNO₃) but will not be studied in the present work. The compositions also differ by the colour producing components and the amount of resin. The combustion of red Bengal flames under air atmosphere produces the highest level of CO₂ yield. It is easily explained by the composition of the firework which contains more carbon than blue and yellow ones. Moreover, it also appears that Blue Bengal flames present the lowest emissions of CO₂ in case of well-ventilated combustion. One more time, it can be related with the composition of the material where carbon will be mainly present in the resin. The emissions of CO, THC, NO, HCl and soot remain very low during the reaction under air atmosphere. When the reacting medium is changed to N₂, fireworks chemical behaviour differs. For the red Bengal flames, the CO₂ yield is significantly decreased by more than half while the CO generation substantially increases. On the other hand, combustion time decreases under N₂ atmosphere. The blue Bengal flame sees a slight decline of the CO₂ yield, combined with a low rise of the CO generation. As the blue Bengal flame seems to contain less fuel, the change from air to N₂ atmosphere in the reaction zone causes minor variation in the combustion gases concentrations. The yellow Bengal flame would represent an intermediate behaviour between the one observed with the red and blue compounds. The productions of THC, NO and soot do not seem to be affected when reaction takes place into a N₂ atmosphere rather than in air. No significant differences are measured regarding the combustion times.

Table 2. Yield of various combustion products from burning of sparkles and Bengal Flames (BFl) under air atmosphere.

Sample	Sparklers	Blue BFl	Yellow BFl	Red BFl
Atmosphere	Air	air	air	air
Initial Mass (g)	15.88	16.32	14.91	15.13
Total Mass Loss (g)	3.73	13.52	12.55	12.1
% Mass Lost	23.5	82.84	84.17	79.97
Combustion Time (s)	74	232	122	220

Combustion Products	Yield (g/g of fuel)			
CO₂	0.528	0.368	0.461	0.627
CO	0.041	0.005	0.005	0.007
soot	0.004	0.049	0.022	0.014
THC	0.002	0.001	0.001	0
NO	0.106	0.003	0.003	0.002
HCl	Nm	0.091	0.096	0.076

Table 3. Yield of various combustion products from burning of sparklers and Bengal Flames (BFl) under N₂ atmosphere.

Sample	Sparklers	Blue BFl	Yellow BFl	Red BFl
Atmosphere	N ₂	N ₂	N ₂	N ₂
Initial Mass (g)	12.48	16.69	14.7	15.45
Total Mass Loss (g)	2.61	14	12.13	12.93
% Mass Lost	20.91	83.88	82.52	83.69
Combustion Time (s)	134	210	120	188

Combustion Products	Yield (g/g of fuel)			
CO₂	0.242	0.336	0.387	0.258
CO	0.164	0.019	0.056	0.178
soot	0.004	0.04	0.023	0.016
THC	0.016	0.003	0.002	0.006
NO	0.121	0.002	0.003	0.002
HCl	nm	nm	nm	nm

*nm: non measured

Figure 2 and Figure 3 represent the molar fractions of the major gases produced during the combustion respectively for sparklers and yellow Bengal flames. Sparklers seem to show a different kinetic depending on the reacting atmosphere. Varying the O₂ concentration seems to slow down the reaction. Under air atmosphere, apart for NO₂, combustion gases begin to be produced simultaneously (about 26s). A first slope between 26 and 40s is then followed by a plateau between during about 4s. Afterwards, CO₂ and NO rise again, while CO stays relatively constant and THC decrease. At 76s, a maximum is recorded for CO₂ and NO which then begin to drop off as does CO. At the same time, the molar fraction of NO₂ begins to increase to reach a maximum peak at 86s which corresponds to a sharper decrease of CO molar fraction. For the reaction under N₂ atmosphere, the dynamics are relatively similar between the combustion gases, apart again for NO₂ which starts every time to be produced during the fall of production of the other species.

Figure 3.a. presents the production of gases from the combustion of yellow Bengal flames. At ignition, the considered species start to be generated. However, different dynamics are observed. CO₂, CO, THC and SO₂ show plateaux which correspond to constant reaction rates. Magnitudes illustrate variations (as observed for CO₂ and CO molar fractions) that are due to the experimental setup. Three fireworks were positioned so that they were supposed to ignite one after the other. However, in the present experiment, two ignited simultaneously creating the divergence observed in the plateaux levels but this has no influence for interpreting the results. NO and NO₂ present peaks at the ignition of each Bengal flame which are followed by fast decrease of the concentration of these two species. Reaction last for about 2 minutes.

Under N₂ atmosphere, the combustion reaction time is relatively similar to the one under air. Same dynamics were noticed but of different magnitudes. The CO₂ production decreases while more

incomplete combustion products (CO, THC and SO₂) are generated. Concerning nitrogen oxides, peaks appear again for a lapse of time after ignition with the same order of magnitude as for the tests carried under air.

From the previous experiments, it emerges that the major combustion gases produced are CO₂ and CO. Reducing the O₂ concentration leads to a less complete combustion. CO but also THC or SO₂ become more increasingly generated while the CO₂ concentration decreases. However, burning still occurs. For the Bengal flames, it seems that constant generation rates are observed for CO₂, CO, THC and SO₂. On the other hand, peaks of NO and NO₂ are observed at ignition for the yellow flames.

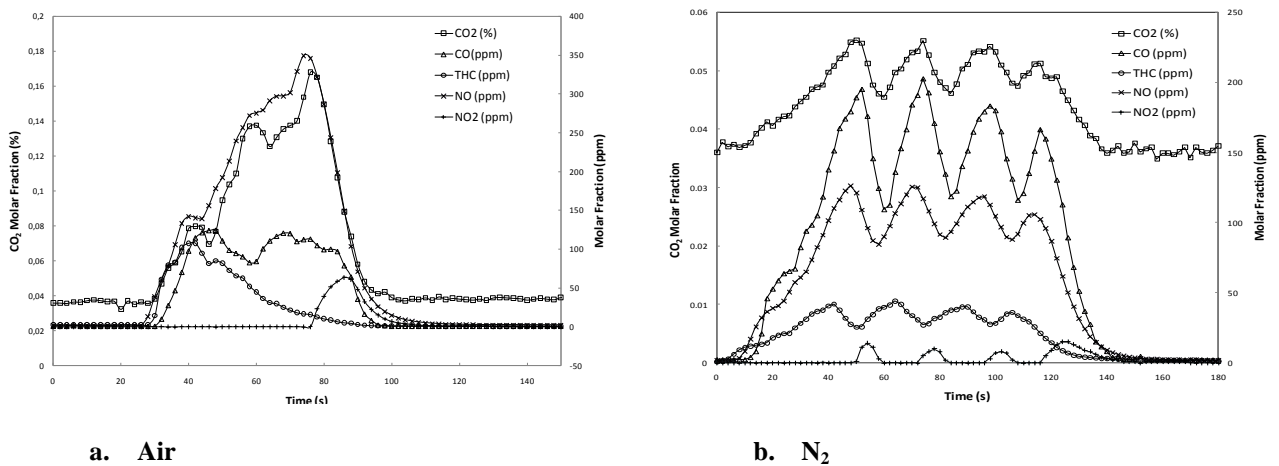


Figure 2. Combustion of sparklers under Air (a) and N₂ Atmosphere (b).

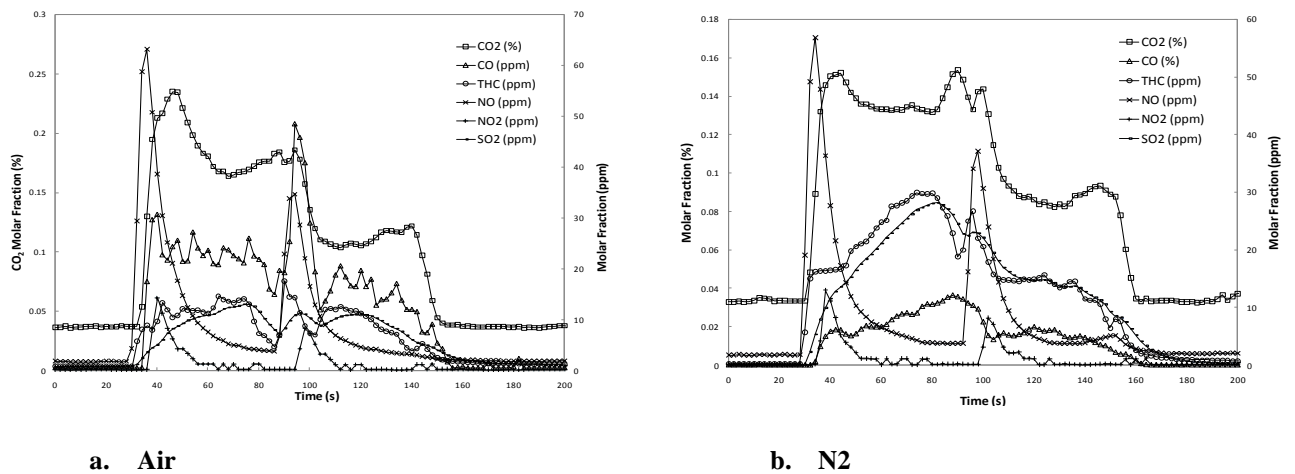


Figure 3. Combustion of Yellow Bengal flames under Air and N₂ Atmosphere.

Discussion

Pyrotechnics present the peculiarity to carry an oxidizer and do not require O₂ from the air to burn. The possibility of a fire even in low O₂ concentration conditions is not negligible. They can be found during a hazard, for example in a vitiated atmosphere. To explore this specificity, combustion experiments have been carried out in air as well as in N₂ atmosphere. Results have been compared to

underline the variations in the production of combustion gases depending on the reacting atmosphere. Ignition occurred for every tested. The results initially expected would be similar to the ones that would be observed for conventional fires in under ventilated conditions (i.e. fuel-rich). The combustion tends to be largely incomplete; the CO₂ generation significantly decreases while the opposite effect is observed for the CO, THC productions.

If discrepancies appeared when the reacting medium is changed, the variations in terms of production of combustion gases are not always significant. If CO₂ yields are divided by 2 and 2.5 respectively for sparklers and red Bengal Flames, they only slightly decrease for yellow and blue Bengal flames. Similar comments can be done for the CO generation. In the case of the Bengal flames, they all enclose the same oxidizer, potassium chlorate. It highlights a critical dependency on the composition of the firework. The fuel, the binder and quantities of each compound are as dominant factors as the oxidizer to understand the material behaviour.

First consider the sparklers. When KNO₃ decomposes, it releases O₂. Freeman [7] suggested the following reaction:



At the same time, the fuel decomposes into volatizes which will first form CO by reacting with radicals and in a second time, CO will be oxidized into CO₂ [8]. We first observe an increase in the generation of CO₂, CO and THC. Then, the CO and CO₂ reach a plateau. This instant correspond to the start of decrease of THC which present after a different dynamics. A hypothesis can be that hydrocarbons are oxidized to produce CO. The more O₂ is released from the oxidizer or present in the air, the faster goes the reaction HC + free radicals → CO. As CO is produced, it will be oxidized into CO₂ if enough O₂ is present in the medium. If the reactions HC + free radicals → CO and CO + ½ O₂ → CO₂ have close reaction rates, it can explain the constant level of CO and the increase of CO₂. Moreover, as HC are more consumed, there concentration decreases. Under N₂, the only O₂ supply comes from the oxidizer. HC will react with radicals to form CO. Because less O₂ is available, less CO will be oxidized into CO₂.

Under air or N₂, CO₂ and NO seem following the same dynamics. An assumption is to relate this behaviour with the decomposition of the oxidizer. Blackwood and Bowden [9] [10] assume the following reaction to explain the formation of NO during the combustion of black powder:



Sparklers composition is very similar to the one of black powder. To verify this hypothesis, it could be interesting to measure in the future the production of K₂SO₄. Nevertheless, the formation of CO₂ is mainly dependent on the O₂ supply from the oxidizer. The production of CO₂ is related to the reaction rate of the KNO₃ decomposition. The similarity of both dynamics tends to the assumption developed previously.

The combustion time is almost doubled when changing the reacting medium from air to N₂. Temperature measurements in the quartz tube are lower in the N₂ medium. Then, reaction rates are then lesser. It has been previously noticed that the same percentage of mass was burnt, which explains the longer duration of the reaction.

Independently of the reacting atmosphere, the production of NO₂ starts once, the generation of CO₂, CO and NO decreases. A possible explanation is to consider that NO₂ is produced by the oxidation of NO when remaining O₂ is present. Several assumptions have been introduced in order to try to understand the decomposition of sparklers. Additional tests would be necessary to verify them.

Moving to Bengal flames, mass losses and combustion time are relatively constant under air or N₂ atmosphere. Temperature in the tube does not vary significantly, so reaction rates may be considered constant. CO₂ and CO generation rates are sensibly constant. The specimens are contained in paper tubes. Their main aim is to burn with the same intensity for a given time then, once ignited, the spread rate of the reaction front needs to be constant. It can be achieved if its area is fixed. Moreover, the oxidizer allows mixing of volatiles and O₂ in the medium while air is only in contact at the surface. This can explain the small variation in CO₂ generation when changing the reacting atmosphere. The oxidizer is the main supply in O₂ but the O₂ concentration has clearly an effect on the rates of combustion gases generated.

Peaks of NO and NO₂ are observed for every ignition of a yellow Bengal flame. The formation of NO and NO₂ can only be due to oxidation of N₂, as the mixture does not contain nitrogen. This reaction occurs for high temperatures. Nevertheless, measurements of the temperature above the flames show consequent peaks at ignition. The hypothesis is that high energy is released at ignition inducing high temperatures allowing the formation of NO and NO₂ following the reactions [11],



Reactions (3) and (4) also imply that O₂ is available for N₂ oxidation. It could be another reason explaining the decrease of NO and NO₂ after ignition. The assumption of a large release of energy at ignition could be verified by estimating the heat release rate (HRR). As previously mentioned, the FPA is equipped to allow calculating the HRR based on OC or CDG calorimetry. The estimations realised show significant discrepancies between the methods. Because of the presence of the oxidizer in the material, the OC calorimetry does not appear to be relevant in the current study because based on the measure of the consumption of O₂. Applying this technique would suppose introducing a correction in the calculation accounting for the internal supply of O₂. On the other hand, the CDG calorimetry relies on energy constants with large uncertainties. No accurate estimation of the HRR can be obtained. A solution would be to measure the heat of combustion of the material by means of a bomb calorimeter.

Conclusion

Lessons from the past have demonstrated that hazards related to the storage and the transport of pyrotechnics were real. It is of great importance to be able to identify and quantify the risks associated with these materials that will have to fulfil in a near future more stringent sustainability criteria. To achieve these objectives, relevant tests have to be defined. The FPA (developed primarily for the fire science community needs) has already shown its ability to characterise the flammability of materials in case of “conventional” fires. Pyrotechnics present the singularity to carry an oxidizer and are then able to sustain flaming even without O₂ from the air. Conditions of low level of O₂ concentration can be encountered during a fire and it is critical to be able to understand how behave pyrotechnics in these circumstances. The advantage of the FPA is that experiments can be carried under variable O₂ concentration levels.

Tests have been carried out with two different types of pyrotechnics: sparklers and Bengal flames. The reacting medium has been varied from air to N₂ atmosphere to observe the effect on the combustion gases generation. Ignition occurred for every test, highlighting the role of the oxidizer as the main reaction partner. Under N₂ atmosphere, it emerges that the emission of CO₂ decreases while the CO increases. However, the magnitude of this behaviour is dependent on the nature of the mixture. If it was significant for sparklers and red Bengal flames, it was noticeably lower for blue and yellow Bengal flames. Moreover, tests showed that independently of the atmosphere, generation rates of the major species were relatively constant. Different reactions have been underlined for the formation of nitrogen oxides. It is essential to be able to identify such compounds given the pollutant nature of NO₂.

The FPA was able to access essential information about the chemical and thermal characteristic of a material. It is able to identify the major species generated during the combustion and their evolution with the O₂ concentration. However studying in depth the combustion toxicity related to pyrotechnics is a complex issue given the diversity in the chemical processes involved. Being able to estimate the HRR and identify more species taking place in the combustion reaction will be a next step in order to better quantify the toxic potency of fireworks. In the future, other tests will be realised coupling FPA and FTIR to better characterise emissions of toxics. First, black powder will be inerted with alumina to slow down the combustion and allow a chemical and thermal characterisation. Results will be compared to previous studies ones. In a second time, other pyrotechnics materials, such as pyrotechnic stars, comet flares or smoke powders, will be tested based on a similar procedure in order to attempt defining relevant test methods.

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