An experimental study of the thermal performance of a novel intumescent fire protection coating

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Abstract

The thermal performance of a novel intumescent coating was investigated at a laboratory scale. A combination of small and large-scale tests was performed in order to fully understand the behavior of the coating. For small-scale testing, experiments were conducted using thermogravimetric analyses. These experiments were run at several heating rates in a nitrogen atmosphere. The results showed that the thermal degradation of the coating occurred in different stages, and, the main mass loss took place around 300°C. Furthermore, the current work showed that oxygen doesn’t exert any significant effect during the early stages of degradation of the materials; however, its interference can be noted past the attainment of the peak value for mass loss rate curve.

For large-scale testing, the experiments were carried out in a cone calorimeter using a stainless steel plate as a platform to support the test specimen. The back surface temperature and expansion height of the intumescent coating were measured as a function of time. Several factors such as heat flux, distance to cone heater and coating thickness were also investigated. The results showed that the normalized expansion height of intumescent coating was consistent at different heat flux levels. Hence the expansion of the coating can be considered to be dependent only on the mass loss rates and not the value of the external heat flux.

Also, results from the cone tests, permit the formulation of an experimental protocol for evaluating of the thermal shielding efficiency of the intumescent coatings. The results
showed that the data obtained using a cone calorimeter with 2.5 cm of distance cannot be compared with other distances, such as 4 or 6 cm. The present work also showed that the values of the relevant parameters did not differ significantly at distances to the cone heater above 4 cm.

In a second evaluation, the new intumescent coating was applied to polyurethane and Gypsum boards, for study using cone calorimetry. The use of the coating led to a decrease in the peak of heat release rate for combustion of polyurethane. The application of a coating layer can be used to decrease the overall requirement of thickness of the Gypsum board without compromising its thermal insulation performance.

**Keywords** Cone calorimeter; Thermogravimetric analysis; Fire-protection; Intumescent coating; Temperature profiles; Thermal shielding.

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1. Introduction

The protection of construction materials against fire is an important issue in the construction industry, especially when newer materials for construction are being introduced into the sector [1]. Some building materials, such as steel, are non-combustible. However, the structural properties of steel change at sufficiently high temperatures, induced by being heated in a fire, which can be lead to the failure of building structures where steel is used as the primary load-bearing component [2].

There are several methods to reduce the detrimental effects of fire on the structural components, such as the application of thermal shielding coatings, cement-based sprays and
use of batten materials [3]. One of the most efficient ways to increase the fire resistance of construction materials is through the application of an appropriate intumescent coating. This technique can be applied to different backing materials, such as metals, polymers, steel, textiles, wood, etc. [3-4]. Intumescent coatings generally form a carbonaceous char layer during the degradation and expansion. This char layer can often act as an efficient thermal shield between the heat source and the materials [5]. However, the ultimate structural stability of the char layer depends on the temperature profiles and the amount of oxygen present in the system. Typically, intumescent coatings are expected to maintain the integrity of structural materials (such as steel) for one to three hours, during which the surrounding temperature exceeds 1100 °C and O\(_2\) concentration drops to a very low level. Therefore, the thermal shielding efficiency of intumescent coatings is of great importance [1]. However, there is an influence of heating rate. If too fast, the heat wave could reach the materials before any development of intumescence.

There are several approaches for estimation of the thermo-protective properties of intumescent coatings, such as standard furnace and natural fire tests [5]. Nevertheless, the assessment of the thermal insulation performance of intumescent coatings is not an easy task given the cost and time required for a proper classification and approval rating of the materials. Standard testing regimes available for building construction materials often include full-scale experiments. However, it is a common practice to test the efficacy of intumescent coatings on stainless steel plate by employing laboratory-scale techniques. For example, apparatus such as a thermogravimetric analyzer and cone calorimeter have been extensively used in order to test the protective effectiveness of various intumescent coatings at a laboratory scale [1, 6-10].

The results obtained from TGA tests may not be applied to real-life fire situations. However, the flexible heating regimes on milligram size samples of a solid material can help to elucidate the various thermal and thermo-oxidative processes that may occur as the materials is heated. Moreover, these tests can also be used to support computation of relevant kinetic parameters. On the other hand, the study of the evolution of mass loss, as a function of temperature, through TGA tests does not necessarily allow a complete and detailed evaluation of all the underpinning thermal reactions that occur [11]. Furthermore, given the relatively very small size of the test sample, it can be only considered behaving as a ‘thermally thin’ material, and thus cannot be considered as a representative test specimen.
However, the thermograms obtained are useful as reliable indicators regarding the degradative behaviours of the test materials upon progressive heating from an external source. In contrast to a thermogravimetric analysis, a cone calorimeter will definitely yield better degradation and combustion profiles for materials, and therefore it is considered to be the standard laboratory-scale test apparatus [12]. It also measures the evolution of mass loss, as well as mass loss rate as a function of time. The other useful parameters from cone calorimetric runs include: time-to-ignition, heat release rates, total heat released, CO, CO$_2$, and smoke yields, etc. The conditions of thermal degradation are more realistic for both thermally ‘thin’ and ‘thick’ materials. Moreover, with larger scale heat fluxes, and heat and mass transfer phenomena, the behaviors of materials in a cone calorimeter can be considered as a better indicator of their corresponding behaviors in real fire scenarios. However, it should be noted here that the actual size and orientation of real fire loads, say in enclosure fires, cannot be realized during the cone measurements, and that convective mode of heat transfer and any heat losses, facilitated through the sample holder, are largely ignored.

Generally, the results from cone calorimetric investigations cannot be easily compared. This can be mainly attributed to the difference in the test configurations, such as the distance between sample and heater, thickness of the coating, nature of the backing materials (plate), heat flux, and to the actual nature of the parameters that are measured. For instance in one investigation, the profile of temperature at the interface between the intumescent coating and the metal plate at a constant value of heat flux (30kW/m$^2$) with 1 mm thick of intumescent coating has been reported [7]. Here it should be also noted that the samples in the sample holder were insulated with four layers of 3 mm thick Cotronics ceramic paper. However, an attempt of compare results from a closely related study was not possible because the thickness of the coatings were quite different (in the range 5-15 mm) [8].

In the present work, the fire performance of a new intumescent coating was investigated using thermogravimetric analyses in order to determine the behavior of the intumescent coating, as a function of increasing temperature.

The mass and mass loss rate were quantified during cone calorimetry experiments. In addition, cone calorimetric data were obtained at different heat fluxes, and the intumescent height was continuously quantified during the runs. In parallel, the temperature of the back surface was measured using a k-type thermocouple.
In order to gauge the benefits of using the new intumescent coating, experiments were also conducted using different backing materials (polyurethane plaque: 240 kg/m$^3$, 25 mm thickness and Gypsum boards: with thicknesses of 15, 12 and 9 mm) at a pre-set value of heat flux of 50kW/m$^2$. This value is widely used as standard in order to compare the behavior of materials in cone calorimetric experiments [13]. An experimental protocol for evaluating the fire protective properties of intumescent coatings, which accounts for the influence of many parameters which can affect the thermal performance of the coatings, has been developed based on results obtained in this study.

2. Experimental set-up

2.1. Materials

Steel plates ($100 \times 100 \times 5$ mm) were coated with an intumescent material. This was a new water-borne intumescent coating developed and supplied by FARBE SPA Company, Milan, Italy. The salient characteristics of the intumescent coatings are listed in Table 1. In order to improve the quality of the coating, potassium chloride, hydrogenated castor oil, potassium ferrocyanide and hydroxypropyl methylcellulose were added to the coating. These ingredients are incorporated, primarily, to improve the uniformity of the coating as well as the adhesion between the coated layer and the materials. All operations such as importation of the materials and the preparation of the coating were completed by FARBE SPA Company (Italy).

Polyurethane and gypsum boards were used as the base for the coatings in order to assess the benefits of using intumescent coating on different materials. The polyurethane samples ($100 \times 100$ mm; 240 kg/m$^3$) were supplied by VA-Q-TEC AG. According to the manufacturer, the samples contained no fillers. The total thickness of polyurethane (240 kg/m$^3$) samples was around 25 mm, with an initial mass of about 60 g. The supplier would like to develop vacuum insulated panels with the polyurethane as a cover for the core.

The gypsum board samples ($100 \times 100$ mm), and with various thicknesses of 15, 12 and 9 mm were obtained from KNAUF-UK. The initial mass for a 15 mm thick board was about 104 g.

2.2. Thermo-gravimetric (TGA) analyses

To characterize the thermal behavior of the materials at mg balance, TGA experiments in nitrogen and air were carried out using a Mettler Toledo TGA instrument, with ca. 10 mg
samples, at four heating rates of 5, 10, 15 and 20°C/min, over a temperature range from 20°C to 1000°C. Samples were ground to a fine powder using a mechanical grinder prior to the runs. A high resolution digital balance was used to ensure accuracy of the initial masses of the samples (ca. 10 mg in each case). The TGA furnace and the balance were flushed with nitrogen at a flow rate of 50 ml/min and 100 ml/min respectively. A test was performed with an empty pan in order to identify and correct the buoyancy effects of the furnace; this baseline run was subtracted from the mass measurements of the samples that were undergoing the thermal decomposition.

2.3. Fire testing

The combustion properties of the materials were measured using a cone calorimeter in conformance with the standard, ISO 5660-1 [12]. Samples were tested at different heat fluxes, and ignition was effected using a spark igniter. The operating principle of the cone calorimeter (oxygen consumption calorimetry) is described in detail elsewhere [14-16]. The sizes of the samples were ca. 100 mm × 100 mm, with varying thicknesses. The exhaust hood air flow rate was maintained at 24 l/s. The temperature of the back surface was measured by a k-type thermocouple.

It is generally known that the performance of intumescent coating depends on both the height and also the mechanical integrity of the char layer produced. In order to follow the degree of expansion, the height profiles of the coating were captured using videography. The video images were then processed by using free software (Kinovea) to track the expansion process and the expanded coating height profile in detail [17-19].

2.4. Sample holder

In this work, the cone calorimeter tests were conducted by using a specific sample holder as reported before [20-21]. Figure 1 presents the sample holder used and the position of the thermocouple. The samples in the sample holder were insulated with four layers of 3 mm thick Cotronics ceramic paper (density = 192 kg/m³; heat capacity = 1046 J/kg.°C and thermal conductivity = 0.06 W/m.K at 260 °C). The sample holders were dried in an oven for 24 hours (at 100°C) and then kept in a desiccator before each experiment.

3. Results and discussion
3.1. TGA tests

The mass evolutions of the intumescent coating, ICWB, under inert atmosphere (N\textsubscript{2}) are plotted in Figure 2, as a function of the temperature, for different heating rates (5, 10, 15, and 20 °C/min). Under nitrogen, the following changes in the curves of mass evolution as a function of temperature (in Figure 2) can be noticed:

The thermal decomposition is mainly observed between 200 and 500 °C, however, the thermal decomposition can occur in different stages [22-29].

The general profiles of the thermograms are very similar; however, the curves are found to be slightly shifted towards higher temperature ranges with increments in the heating rate [28-29].

From the ambient temperature to 100°C, around 5% of the initial mass is lost, possibly owing to the escape of moisture and other low molecular weight species.

Between 100 and 300 °C, a mass loss around 10% of the initial mass is observed.

Between 300 and 400°C, a mass loss around 25% of the initial mass is observed.

Between 400 and 550°C, around 15% of the initial mass is found to be lost.

Between 550 and 900°C, the mass remained constant.

Between 900 and the end of the test, a mass loss around 15% of the initial mass is observed, and this can be attributed from degradative reactions occurring to the relatively stable components in the mixture.

At the end of the tests, a residue of about 30% of the initial mass of the samples remained in the crucible.

Through a close evaluation of thermograms, in Figure 2, a clear identification of all the steps in the thermal activation and degradative pathway of the intumescent coating under inert atmosphere is not easy, as the subtle changes in the slope of the thermograms are not very conspicuous.
Figure 3 shows the MLR curve evolutions as a function of the temperature at the different heating rates under inert atmosphere.

Evidently, it can be observed that the MLR profiles are quite similar, qualitatively, regardless of the value of the heating rate employed. Nevertheless, the slope of the curves and the peak intensities strongly depend on the heating rate chosen, i.e. the higher the heating rate is the higher is the peak intensity. This observed behavior is in agreement previous results reported in the literature [22-23]. The results presented in Figure 3 are also in consistent with previous investigations which demonstrated that the thermal degradation of intumescent coating generally occurs in many different stages [17, 24-27]:

Stage 1: Evaporation of low molecular weight volatiles: no significant expansion (ambient temperature - 130°C).

Stage 2: Softening: no significant expansion (130-180°C).

Stage 3: Decomposition of the blowing agent: a rapid expansion of coating (between 180 and 300°C).

Stage 4: Formation of char: where maximum rate of mass loss occurs (300-500°C).

Stage 5: This stage occurs at higher temperatures, where decomposition of char happens (500-1000°C).

In the present study, the thermo-oxidative decomposition behaviours of the intumescent coating, for heating rates from 5 to 20°C/min, under air atmosphere were also evaluated. This will supplement information to previous data obtained under inert atmosphere, and will also enable to identify the thermo-oxidative reactions and thus to describe the influence of the oxygen on the thermal decomposition mechanism of the intumescent coating.

Figure 4 presents the evolutions of the normalized mass under air atmosphere, as a function of the temperature, for the four heating rates employed. Even though the general profile of the curves is similar, they are found to be shifted towards higher temperatures [28-29].

Under air, the following changes in the profiles of the curves denoting mass evolution as a function of temperature can be noticed (Figure 4):
All the curves are nearly identical for the different heating rates. The thermal decomposition mainly occurs between 200 and 800 °C.

From the ambient temperature to 100°C, around 5% of the initial mass is lost, and this can be attributed to the volatilization of low molecular weight species.

Between 100 and 300 °C, a mass loss around 10% of the initial mass is observed.

Between 300 and 400°C, a mass loss around 25% of the initial mass is observed.

Between 400 and 600°C, around 15% of the initial mass is found to be lost.

Between 600 and 800, a mass loss around 15% of the initial mass is observed.

Between 800 and 1000°C, the mass remained constant.

At the end of the tests under air, a residue of about 30% of the initial mass of the samples remained in the crucible.

A detailed observation of all the reactions occurring, in terms of the mass loss, during the thermal decomposition of intumescent coating under air atmosphere is represented in Figure 5. Here it should be also noted that the MLR profiles are quite qualitatively similar regardless of the value of the heating rate studied. The oxidation of char is properly visible over 500°C.

3.2. The influence of the atmosphere

In order to identify the influence of the atmosphere on the thermal behavior of the intumescent coating, a comparison of the mass and of the mass loss rates, under nitrogen and in air for heating rate of 5 and 20°C/min (Figure 6).

Through a comparison of the mass and the mass loss rate under nitrogen and in air, as presented on Figure 6, the following can be observed:

The general profiles of the mass and mass loss rate curves are quite similar for the two atmospheres for temperatures lower than 400°C.

Between 400 and 500°C, the mass loss rate hardly increases under nitrogen (with an abrupt change in slope), and the mass loss rate reached its maximum value at 475°C (where as in air,
the mass loss rate still decreases). In order to explain the result obtained between 400 and 500°C, an oxidative reaction could be considered to have occurred under air [17].

Between 500 and 800°C, an additional peak of MLR is observed under air (i.e. corresponding to the oxidation of char), while no corresponding mass loss peak is observed under nitrogen.

When the temperature is above 800°C, a mass loss is observed under nitrogen atmosphere only.

Overall, the nature of the prevailing atmosphere does not seem to affect the thermal degradation of the material for temperatures less than 400°C.

The choice of nitrogen for the TGA temperatures was chosen following some previous studies [24, 28-29]. These investigations showed that air is not involved in any reaction in the early stages of expansion of the coating. In fact, the effects of the atmospheric oxygen mainly occur when the temperature was greater than 540°C [17, 24]. Thus, no significant difference was observed between degradation of intumescent coatings whether in air and nitrogen at the early stages [29], which also was found to be true in the present work.

However, the current work showed that oxygen doesn’t exert any significant effect during the early stages of degradation of the materials; however, its interference can be noted past the attainment of the peak value for mass loss rate curve.

3.3. Cone calorimeter experiments

a) Distance to the cone heater

The heat flux received on the surface of the sample can be adjusted either by setting the temperature of the cone heater, or by varying the distance from the cone heater to the sample. This distance is generally specified as 25 mm in the standard [12]. In fact, the distance between the sample and the cone heater is not a critical factor under normal circumstances, but in the case of an intumescent coated surface, the expansion of intumescent coating leads to a decrease in the amount of the heat energy received at the surface and the edges of the stainless steel plate.

Figure 7 presents the development of the back surface temperature of the coated stainless steel plate with time, at 50 kW/m², for different distances of 25, 40 and 60 mm. The ISO 5660-1:2015 [12] mentioned that intumescent samples that they contact the park plug prior to
ignition or the underside of the cone heater after ignition shall be tested with the separation of 60mm between the base plate of the cone heater and the upper surface of the specimen. In this case the calibration shall be performed with the heat flux meter positioned 60 mm below the cone heater base plate [12]. Due to the prevailing favorable view factor, the heat flux is assumed to be uniform at the surface of the sample at 60 mm [30-32].

The results were compared to the corresponding back surface temperatures of steel without coating. As can be seen, the temperatures are similar at the beginning of the test for all the cases. After about 100 sec, the temperature kept on increasing up to 600°C for steel plate without coating. In addition, the slopes of the temperature profiles for the coated samples were found to decrease with time. It seems that the temperatures for the coated samples are not related to the distance between the cone heater and the sample until about 400 sec. The temperature attained at the standard distance (25mm) is found to be higher than those for the other distances (40 and 60mm). Apart from this feature, the results with regard to the temperature values for distances 40 and 60 mm are almost similar. In fact, for the smaller distance (25mm), the intumescent coating was found to be expanded more than 25mm, thus a part of the intumescent coating became percolated into the cone heater thus effecting a markedly different behavior as compared to the other two cases (40 and 60 mm). The difference can be explained by the uniformity of the radiant heat flux striking the intumescent material’s surface in the cone at the higher distances [30]. In addition, in a previous investigation [31], it was shown that the temperature and heat input distribution on the exposed surface of a specimen in a calorimeter can be affected by variations of position or size of the specimen, as it undergoes volumetric progressions in cone calorimeter tests. Hence, the amount of radiant heat absorbed by the top and side surfaces of the recipient can be also vary during the whole duration of the runs [32]. Therefore, the variations of the results obtained in the Figure 7 can be attributed to the variations in the heat fluxed received by the surface. In fact, the amount of heat flux received by the surface will significantly vary, owing to the extensive swelling of the intumescent coating, during the experiment.

In order to quantify the variation of heat fluxes in the horizontal direction, a calibration of heat fluxes was performed, at different vertical and lateral locations, using a Gardon gauge. The experimental data are presented in Figure 8 along with the theoretical values calculated using the ‘view factor’ following a literature precedent [30]. The measured experimental values showed a good agreement with those of the predicted ones (Figure 8):
- From 60 to 27 mm in the centre.

- From 60 to 12 mm at 2cm from the centre.

- From 60 to 12 mm in the corner, if an average value from both tests is considered.

For example: the nominal heat flux is 50 kW/m² at 60 mm, and the measurements here are in good agreement with the calculations. The heat flux at the centre increases from 50 to 72 kW/m² when the distance changes from 60 to 12 mm. Furthermore, the heat flux decreases away from the centre for most of the heights considered, except for the highest location where an opposite behaviour was noted.

b) Heat fluxes vs. surface temperatures of the stainless steel plate

The aim behind making an intumescent coating is to increase the time taken for the steel to reach a higher temperature, for example 550°C [33]. Significantly high temperatures have an influence on the mechanical properties of steel, possibly leading to structural failure, resulting from substantial loss of its mechanical strength. As presented in Figure 7, the temperature of steel without coating, at 50kW/m², can exceed 550°C. This is depending on the emissivity of the steel (around 0.6 in this work).

Figure 9 presents the evolution of the back surface temperatures of the coated steel plate as a function of time at heat fluxes of 30, 40, 50 and 60kW/m². Note here that the experiments were carried out at two values of distance between the bottom of the cone heater and the sample (i.e. 25 mm and 60 mm).

It is rather difficult to measure the evolution of the thermal properties for the intumescent coating, such as the effective thermal conductivity, directly during the experiments (in cone calorimeter and other larger scale tests). Hence, the thermal properties are generally assessed by measuring the backing material’s temperature. As can be shown in Figure 9, the evolution of the back surface temperature for the steel plate is related with the distance between the bottom of cone calorimeter and the sample. The temperatures measured at distance 25 mm are higher than the temperatures obtained at distance 60 mm. The deviation between the two groups of measurements is also found to be related to the heat flux. For example, the temperatures after 1200 sec at 30 and 60kW/m² are 300 and 425°C, respectively, for 25mm case. While the temperatures under the same conditions are 275 and 320°C for the higher
distance (60 mm). For both cases of distances, two regimes can be clearly observed. The first
one is before the change in slope, where the temperature of the steel rises quickly and almost
linearly with time as the sample directly absorbs the heat from the cone calorimeter. Then in
the second step, the steel temperature continued to increase at a lower rate due to the
appearance and development of the intumescent coating. Here it can be also noticed that the
point when slope is changing in a step-wise manner is almost constant. The intumescent
coating seems to reduce the quantity of heat received from the cone to the steel plates
significantly. Therefore, compared to the steel plate temperatures without a coating, the
intumescent coated surfaces appear to possess good thermal protection. It was also noticed
that there was a significant time delay for the temperature to attain the maximum value when
the experiments were also conducted at 25mm distance.

c) Heat flux vs the height of the intumescent coating

The expansion of intumescent coating when exposed to a heat flux is rather complex,
involving a heterogeneous mixture of gas, liquid and solid phases, and that most of the
accompanying variables are extremely difficult to measure [17]. In the present work, the
heights of the expanded surface layers were measured in order to understand the effects of the
external heat flux on the behavior of the intumescent coating. As mentioned earlier, the
obtained video images were treated by free software named Kinovea with a view to tracking
the expansion processes and the evolution of the height profiles of the expanded coatings [17-
19]. By using the video images and the numerical code, it was also possible to measure the
evolution of the intumescent heights as a function of time for the stainless steel plate.

When exposed to 30 kW/m² heat flux, the intumescent coating started to produce smoke at 65
sec, while releasing of smoke began earlier for tests at the higher heat fluxes. The expansion
of the coatings began at about 110 sec, and kept on increasing until the end of the tests (2000
sec: 28 mm) at 30kW/m². Here, the maximum temperature of the stainless steel plate was
observed to be 274 °C. For the higher heat fluxes, flashing of flamelets was observed on the
top of intumescent coating. The expansion began at 50 sec and 45 sec at 50 and 60kW/m²
respectively. At 635 sec, the height was found to be 27 mm for 50 kW/m² flux value. During
time 635 sec to 1410 sec, the height remained almost the same, possibly as the decrease in
height caused by oxidization was more or less balanced by the increase in height owing to the
expansion. After about 1410 sec, the height slowly expanded up to 28 mm at 1920 sec under
60kW/m², and the expansion in height in this case kept increasing from beginning to the end of the test and attained a maximum value of 36 mm.

Figure 10 presents the evolution of the intumescent coating height as a function of time at different heat fluxes. When the distance was 60 mm, reactions of an oxidative nature can be thought to occur after 635 sec and 375 sec at 50 and 60kW/m² respectively.

Properly, it will be highly appreciated to investigate the effect of the heating rate at nitrogen atmosphere using Universal flammability apparatus (UFA) or controlled atmosphere cone calorimeter in order to study the behavior of the coating without oxidation step.

d) The effect of the quantity of quantity (optimization of the thickness of the intumescent coating)

The intumescent coating is usually applied on the surface of interest and the thickness is noted while it is dry. However, in the present work, the amount of coating applied was monitored instead of its thickness. Table 2 presents the different amounts of coatings applied on samples. The temperatures of the back surface as a function of the time at 50 kW/m² for these samples are plotted in the Figure 11.

The results show that the back surface temperature of steel is a function of the quantity of the applied. A large difference in the values of the back surface temperature for unprotected steel and the sample S1 can be observed. The variation of the quantity of the coating applied on the steel in range from 500 to 1200 g/m² did not in fact led to a noticeable variation in the back surface temperature of the steel. At 1000 sec, the temperatures were around 360°C and 325°C for 500 and 1200 g/m² cases respectively. Therefore, the amount, 500 g/m², can be considered to be a threshold value in order to achieve an adequate protection of the stain steel plate.

e) Intumescent coating height vs mass loss (The Expansion Function \( f_{\text{exp}} \left( \frac{\Delta m}{m_0} \right) \))

The expansion process includes a series of physical and chemical reactions involving both the solid and gas phases. In practice, the expansion height, \( h \), is usually related to mass loss by a constant ratio [34-36]. Figure 12 presents the relationship between the height and mass loss. Here the expansion height is considered as a function of mass loss, as presented in the following equation:
\[ \frac{h}{h_{\text{max}}} = f_{\exp}(\frac{\Delta m}{m_0}) \]

Where \( h \) is expansion height, \( h_{\text{max}} \) is the maximum height, \( \Delta m \) mass loss, and \( m_0 \) is the initial mass. The value of the maximum height, \( h_{\text{max}} \), is not easy to compute. It can be taken as proportional to the original height of coating, as a fixed value [24], or be different at different heating conditions [17]. Here, \( h_{\text{max}} \) is given as a function of initial mass (\( m_0 \)).

As can be seen from Figure 12, the expansion curves of intumescent coating are consistent at different heat flux levels, hence the expansion of the coating can be considered to be depending on the mass loss.

f) Application of the intumescent coating over different backing materials

In order to verify the thermal performance of the intumescent coating used in this work, the intumescent coating has been applied onto polyurethane (PU) and Gypsum boards (Diamant). The polyurethane is generally used as insulation material in the green buildings [37], and one of the main safety issues in using polyurethane materials is their relatively high combustibility [38]. In this context, it is highly desirable to investigate the thermal performance of polyurethane samples (with and without coating: around 1mm coating). The samples used were a Polyurethane board in a medium density of 240 kg/m\(^3\) and lambda 0.024 W/m.K.

The experiments were conducted in the cone calorimeter at 50 kW/m\(^2\). Table 3 presents the comparison between the results obtained for polyurethane with and without coating. As shown in Table 3, the presence of the intumescent coating increases time to ignition of the samples. As far as the peak heat release rate is concerned, the use of intumescent coating led to significant decrease in its value, with a concomitant decrease in the smoke yield, as compared to the uncoated sample.

Figure 13 presents the heat release rate as a function of time at 50kW/m\(^2\) for un-coated and coated polyurethane samples. As can be seen from the figure, the use of the intumescent coating noticeably decreases heat release rate.

The evolution of back surface temperature is presented in the Figure 14 for the un-coated and coated PU samples at 50kW/m\(^2\) in the cone calorimeter. Here it seems that application of the
intumescent coating leads to an increase in the thermal resistance for the polyurethane material. The back surface of un-coated PU sample attained 600°C after 500 sec, while the back surface temperature of the coated PU coated samples was less than 300°C even after 30 min.

Following on from the local building codes, materials such as Gypsum board may be applied over the rigid foam insulation materials. Normally, a 15-minute thermal barrier, or ignition barrier is required [39-40], e.g. in the case of a French regulation destined for interior insulation walls in public buildings (article AM8) [40]. Some other codes however require a minimum thickness of Gypsum board according the scenario [39]. In this context, in the present study a comparison between the thermal insulation for un-coated and coated Gypsum boards were conducted by using a cone calorimeter at 50kW/m². The Gypsum boards (Diamant) can be used as an element of the internal and external walls [41]. The Fire Euro-class for the board is A.

The application of the coating layer can be used to decrease the thickness of the Gypsum board without compromising its thermal insulation performance. Thus this can lead to the use of a lesser amount of material in the building construction sector, thus paying the way forward for employing light-weight materials. Figure 14 presents the evolution of the back surface temperature for 15 mm un-coated Gypsum board and for coated samples of thickness, 9, 12 15 mm. It can be noticed here that the behavior of back surface temperatures are similar for all the cases until 900 sec is passed.

It can be concluded from the comparison between the results for 15 mm un-coated Gypsum board and 15 mm coated Gypsum board that the use of the intumescent coating increases the duration thermal barrier effect for the material for about by 10 min. For instance, the un-coated Gypsum board without coating attained 200°C after 1000 sec, while the coated Gypsum board attained the same temperature only after 1600 sec. For boards with different thickness, i.e. the 15 mm un-coated Gypsum board and 12 mm coated Gypsum board, demonstrated that use of the light board (less thickness) with intumescent coating led to increase in the duration of the thermal barrier function by 5 min as compared to the thicker board. Furthermore, the back surface temperature for the coated 15mm un-coated Gypsum board and 12 mm coated Gypsum board are quite similar (Figure 15). Thus the use of the coated lighter board (9 mm Gypsum board with the intumescent coating), showed the thermal insulation of an un-coated and heavier board of higher thickness.
Some follow-on large scale fire resistance tests were also conducted on the materials that employed much larger specimens (ca. 3 m × 3 m, and that were relatively thinner). Furthermore, based on the main outcomes from these tests, commercial exploration of materials for internal wall structures (Gypsum boards) with thinner dimensions were commercially explored [41].

Main conclusions

In this study, the thermal shielding efficiency of a new intumescent coating has been investigated. The results obtained using the TGA show that this new intumescent coating possesses a higher heat resistance where the main peak of the thermal degradation occurs around 300°C. The current work showed that oxygen doesn’t exert any significant effect during the early stages of degradation of the materials; however, its interference can be noted past the attainment of the peak value for mass loss rate curve.

The results obtained using cone calorimeter showed that the distance between the sample and the bottom of the cone heater can affect the results of the tests especially the back surface temperature of the materials (i.e. a stain steel plate). Based on the data obtained, the ISO 5660 protocol in terms of the distance from the cone heater to the surface of the sample (i.e. 25 mm) can be modified to accommodate new distances of 40 and 60 mm, instead of 25 mm. It was also established that that the back surface temperatures of the samples were related with the distance of separation of the cone heater and sample surface (i.e. higher values of temperature for the smaller distances).

Higher distances, such as 60 mm, were reported previously [17, 24], especially for numerical investigative purposes. The results so far showed that the deviations in distances from the standard value of 25 mm effect on the output and can lead to get a lower value of back surface temperature as compared to the value for the normal distance of 25 mm. The present work also showed that the values of the relevant parameters did not differ significantly at distances to the cone heater above 4 cm.

In addition, the results for the present study show that the quantity of intumescent coating did not have any significant effect, especially, when it was above than 500g/m². Also, the results showed that the normalized expansion height of intumescent coating was consistent at different heat flux levels. Hence the expansion of the coating can be considered to be dependent only on the mass loss rates and not the value of the external heat flux.
Finally, the effect of intumescent coating used on different backing materials was investigated. The results showed that the use of coating can lead to decrease the heat release rate for polyurethane. Also, the application of the coating layer can result in decrease in the thickness of the Gypsum board without compromising its thermal insulation performance.

Acknowledgements

The authors acknowledge the EU for financially supporting the ELISSA project FP7-2013-NMP-ENV-EeB. The authors also thank Mr W Veighey and Mr Mckee for helping with the experiments.

References

15. V. Babrauskas, Why was the fire so big? HHR: The role of heat release rate in described fires, Fire and Arson Investigation. 47 (1997) 54–57.


40. Arrêté du 6 octobre 2004, portant approbation de dispositions complétant et modifiant le règlement de sécurité contre les risques d'incendie et de panique dans les établissements recevant du public.

Figure 1: The sample holder employed in the present work

Figure 2: Mass loss as a function of temperature for the thermal decomposition of intumescent coating, ICWB, under an inert atmosphere.

Figure 3: Mass Loss Rate as a function of temperature for the thermal decomposition of intumescent coating, ICWB, under an inert atmosphere.
Figure 4: Mass loss as a function of temperature for the thermal decomposition of intumescent coating, ICWB, under air.

![Mass loss graph with temperature range from 200°C to 500°C](image)

Figure 5: Mass Loss Rate as a function of temperature for the thermal decomposition of intumescent coating, ICWB, under air.

![Mass loss rate graph with temperature range from 200°C to 500°C](image)
Figure 6: Comparison of the evolution of the mass and mass loss rate under nitrogen and in air at heating rates of 5 and 20°C/min for intumescent coating, ICWB.
Figure 7: Evolution of the temperatures of the back surface of stainless steel plate at 50kW/m$^2$ for different distances between the bottom of cone heater and the top of steel surface.

Figure 8: Comparison of predicted heat fluxes with measurements for distance between the sample and the bottom of the cone heater, varying from 1.2 to 6 cm. The heat flux is set at 50kW/m$^2$ at 60 mm.
Figure 9: Evolution of the back surface of stainless steel plate at different heat fluxes during cone calorimeter tests.

Figure 10: Evolution of intumescent coating height at different heat fluxes during cone calorimeter tests.
Figure 11: Evolution of back temperature of the steel plate coated by different quantity of ICWB at different heat fluxes during cone calorimeter tests.

Figure 12: Evolution of intumescent coating height as a function of mass loss at different heat fluxes during cone calorimeter tests.
Figure 13: Evolution of the heat release rate for un-coated PU and coated PU at 50kW/m² during cone calorimeter tests.

Figure 14: Evolution of the back surface’s temperatures for PU and coated PU at 50kW/m² in cone calorimeter tests.
Figure 15: Evolution of the back surface temperature for un-coated and coated Gypsum boards at 50kW/m² in cone calorimeter tests.
Table 1: Properties of intumescent coating.

<table>
<thead>
<tr>
<th>Formulation</th>
<th>Solids (%w/w)</th>
<th>Solids (%v/v)</th>
<th>Product concept</th>
<th>Type</th>
<th>Density (g/dm$^3$)</th>
<th>Expansion (ratio)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ICWB</td>
<td>75</td>
<td>69</td>
<td>Water-borne with white color</td>
<td>Halogen free recipe</td>
<td>1.35</td>
<td>1:80</td>
</tr>
</tbody>
</table>

Table 2: The quantity of intumescent coating applied to the samples.

<table>
<thead>
<tr>
<th>Steel samples</th>
<th>Quantity added (g/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>St</td>
<td>0</td>
</tr>
<tr>
<td>S1</td>
<td>250</td>
</tr>
<tr>
<td>S2</td>
<td>500</td>
</tr>
<tr>
<td>S3</td>
<td>700</td>
</tr>
<tr>
<td>S4</td>
<td>1000</td>
</tr>
<tr>
<td>S5</td>
<td>1200</td>
</tr>
</tbody>
</table>

Table 3: Cone calorimeter results for un-coated polyurethane and coated polyurethane (PU) samples at 50kW/m².

<table>
<thead>
<tr>
<th>N</th>
<th>Case</th>
<th>tig(s)</th>
<th>PHRR</th>
<th>$Y_{CO}$</th>
<th>$Y_{CO2}$</th>
<th>$Y_s$</th>
<th>HoC</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>PU</td>
<td>6.5</td>
<td>576.7</td>
<td>0.03</td>
<td>1.86</td>
<td>0.064</td>
<td>16.5</td>
</tr>
<tr>
<td>2</td>
<td>PU coated</td>
<td>16</td>
<td>124.4</td>
<td>0.02</td>
<td>2.36</td>
<td>0.028</td>
<td>12.8</td>
</tr>
</tbody>
</table>

Where: $t_{ig}$: ignition time (s), PHRR: Peak of heat release rate (kW/m²), $Y_{CO}$: carbon monoxide yield (g/g), $Y_{CO2}$: carbon dioxide yield (g/g), $Y_s$: Smoke yield (g/g) and HoC: Heat of combustion (MJ/kg).