

# Cone calorimeter experimental tests report

## **PROJECT PTDC/EME-PME/64913/2006:**

### **“ASSESSMENT OF INTUMESCENT PAINT BEHAVIOUR FOR PASSIVE PROTECTION OF STRUCTURAL ELEMENTS SUBMITTED TO FIRE CONDITIONS”**

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#### **1 - INTRODUCTION**

Passive fire protection materials insulate steel structures from the effects of the elevated temperatures that may be generated during fire. They can be divided into two types, non-reactive, of which the most common types are boards and sprays, and reactive, being intumescent coatings an example.

The intumescent coatings used in the field of the fire protection of steel structures are generally thin intumescent films. They are available as solvent or water based systems applied up to approximately 3[mm]. One problem associated with the use of such systems is the adhesion of the charred structure to the steel element during fire and upon it. It is very important that the char remains in the steel surface to insure the fire protection.

The intumescent chemistry has changed little over the past years and almost all coatings are largely based on the presence of similar key components. The chemical compounds of intumescent systems are classified in four categories: a carbonisation agent, a carbon rich polyhydric compound that influences the amount of char formed and the rate of char formation; an acid source, either free or formed during the reactions upon heating; a foaming agent, usually halogenated or nitrogenated compounds, which, during their degradation release non-flammable gases such CO<sub>2</sub> and NH<sub>3</sub>, [1].

Activated by fire or heat, a sequential chemical reaction between several chemical products takes place. At higher temperatures, between 200-300 [°C], the acid reacts with the carboniferous

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agent. The formed gases will expand, beginning the intumescence in the form of a carbonaceous char.

Different models handle the intumescent behaviour with char forming polymers as a heat and mass transfer problem. Other existing models provide a suitable description regarding the intumescence and char formation using kinetic studies of thermal degradation, accounting the complex sequence of chemical reactions, thermal and transport phenomenon, [2]-[5].

Due to the thermal decomposition complexity of intumescent coating systems, the models presented so far are based on several assumptions, being the most relevant the consideration of one-dimensional heat transfer through material, temperature and space independent thermal properties and the assumption of a constant incident heat flux where the heat losses by radiation and convection are ignored, [3]. Some authors also assume that the thermochemical processes of intumescence occur without energy release or energy absorption, [6].

Anderson et al, [7][8], developed a mathematical model which describes the mechanism of intumescence by considering the mass and energy conservation equations, assuming the heat rate per unit mass generated by chemical reactions mainly at the pyrolysis zone and the heat due to the outgassing of volatile products. The intumescence was accounted considering the mass loss during the process. The model assumes that the transition to the intumescent state occurs at a very thin zone or front and is divided into two regions, the virgin material and the char layers. This model is compared with experimental results performed in steel plates coated with intumescent paints. Later, in [9], Anderson et al present an estimate for the effective char thermal conductivity. The results show that the insulation efficiency of the char depends on the cell structure and the low thermal conductivity of intumescent chars result from the pockets of trapped gas within the porous char which act as a blowing agent to the solid material.

This experimental study is performed to assess the performance of water-based intumescent paints used as a passive fire protection material. These tests were done in a cone calorimeter, in steel plates coated with two different paints, three dry film thicknesses and considering two different radiant heat fluxes. During tests, among other quantities, the steel temperature, the intumescence mass loss and thickness variation were measured.

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### 2 - EXPERIMENTAL TESTS PERFORMED IN THE CONE CALORIMETER

To assess the performance of two commercial water-based intumescent paints a set of experimental tests was performed in a cone calorimeter, see Table 1 and Table 2. The steel plates are 100 [mm] squared and 4, 6 [mm] thick, coated in one side with different dry film thicknesses and tested in a cone calorimeter as prescribed by the standard ISO5660, [10]. Temperatures are measured by means of four thermocouples, type k, welded at the plate in the heating side and at the opposite side, at two different positions.

The samples were weighted before and after of being coated allowing for the initial coating mass. The dry thickness was also measured in 16 different points, being the mean values and the standard deviation presented in the Table 1 and Table 2.

The specimens were wrapped with aluminium foil and between the steel plate and the sample older two silicate plates were used to put the specimen in place and also a thermocouple was placed to measure the temperature variation of them, as presented in Fig. 1.

Although the cone calorimeter test standard specifies that the sample surface for deforming or intumescent systems be fixed by using a retainer frame and wire grid, this “special mounting” was not used. The distance between the sample surface and the heater remained unchanged, at approximately 60 [mm], which means that with the increasing intumescence the top of the sample came closer to cone. This can bring some non uniformity to the heat flux at the sample surface, mainly on the edge, as experienced in the Scharrel et al work, [11].

The temperatures specified at the cone calorimeter electric resistances were 689 and 905 [°C] for the radiant heat fluxes of 35 and 75 [kWm<sup>-2</sup>], respectively.

#### 2.1 - Experimental Results

Fig. 5 to Fig. 8 represent the mass loss of each sample and shows a variation almost linear with time especially for a heat flux of 35 [kWm<sup>-2</sup>]. It wasn't possible to measure the mass loss of the specimens with 6 [mm] thick due to the load cell capacity.

The temperature evolution in a steel plate without protection was also tested to attain the efficiency of this fire protection. The measured temperatures are presented in Fig. 3 and Fig. 4 for a radiant heat flux of 35 [KW/m<sup>2</sup>] and then resetting the cone to 75 [KW/m<sup>2</sup>].

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The steel temperature profiles and at the middle of the silicate plates are reported in Fig. 9-Fig. 20. Measured values from the thermocouples welded on the bottom of the plate are very close to the temperatures at the top. For the same heat flux, the time to reach a same temperature increases with the increase of the dry thickness.

The behaviour is very similar for both coatings, but for all cases the time to reach, for example a temperature of 200 [°C] is always higher when the paint B is used. For these conditions it gives an improved fire protection.

Using discrete frames obtained from the camera during tests and by image processing techniques in Matlab, the intumescence development was measure over time. Fig. 21 to Fig. 32 show the intumescent development (free boundary  $L(t)$ ) for specimens with paint A and B, different thicknesses and radiant heat fluxes. Higher intumescence may be noticed in sample right region coincident to the thermocouples wire position responsible for coating accumulation. The presented values are mean values of four central measures in the distance between the thermocouples.

The figures show that for the lower heat flux the intumescence becomes stable but for the highest it continues to increase. The coating A have a higher expansion at the initial stage compared to the coating B. For longer periods of exposure coating B continues to expand.

### 3 - SUMMARY

This work has presented a set of experimental tests conducted in a cone calorimeter to assess the intumescent coating behaviour when used in fire protection. The intumescence depends on the initial dry thickness and on the incident heat flux.

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