Performance of intumescent coatings in cone calorimeter and open pool fires

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Abstract

Accidental fire is a major concern in terms of safety of infrastructures and human lives. With the technological advancement, several novel methods are developed for minimizing the damages caused by the fire. One of the methods is to paint the base metal/material with fire retardant coatings which can increase the lead time so that economic destruction and loss of human lives can be avoided. In this work, the performance of the intumescent coating (passive type fire retardant coatings) is studied with the help of cone calorimeter and open pool diesel fire as sources of heat. The transient temperature distribution for bare Stainless Steel 310 plate suggests that the cone calorimeter experiments alone cannot suffice for mimicking real life conditions. Comparison of the behavior of the available paints in cone calorimeter and open pool fire confirms that the performance of intumescent coatings in cone calorimeter is very different from that in open pool fire. The safe initial thickness of the intumescent coating is a function of heat release rate of the source. The effective thermal conductivity of the intumescent coatings is evaluated using one dimensional conduction heat equation with constant boundary temperature condition.

Introduction

Metals play a very important role in providing strength to different types of structures. They are the back bone of any kind of construction. Structures like buildings, warships, etc. use heavy amount of steel and aluminum parts for good strength on a long run of life. But accidents like what happened in New York (11/09), wars damages the metallic support used in construction, due to exposure of very high temperature. Such kinds of situations are very likely to happen in naval warships. To avoid either those kind of destruction or to get a lead time to evacuate lives, the structures need to be protected. The protection of the structures can be ensured using fire resistant coatings. The use of intumescent coating is one of the most preferred ways to protect substrate from fire.1,2 The interesting property of an intumescent paint is that it does not affect the thermo-physical or any other property of the substrate. It can be easily applied on to the surface with the help of brush, spray or roller and this paint can be used on any type of solid material like flax boards,3 textiles4 and metals.5 Intumescent paint as the name suggests intumesces when the surface temperature reaches the pyrolysis temperature. There are several theories published in the literature on the mechanism of working of intumescent paints. Deogan et al.6 proposed a one dimensional behavior of the intumescent paint to protect the substrate from fire. When heat is applied to a surface coated with an intumescent paint, it was assumed that no reaction occurred until a critical temperature was reached. Subsequently, an infinitesimally thin front, at which the intumescence occurs, moves through the layer of paint. The temperature at this front is firmly clamped at critical temperature but behind the front (i.e. in the portion where the front has passed) the temperature will rise and ahead of the front (i.e. between the substrate and the front) the temperature must be less than the critical temperature.

Jimenez et al.7 and Gu et al.8 observed that, an intumescent paint is a combination of three compounds; an acid source, a carbon source and a blowing agent. The moment temperature of the surface reaches the critical temperature of the compound, acid source breaks down to yield a mineral acid, then it takes part in dehydration of the carbonization agent to yield the carbon char, and finally the blowing agent decomposes to yield gaseous products, this causes the char to swell in porous form and thus provides the insulation protective layer. The quantity of the three compounds should be selected intelligently based on a set of experiments so that a physical and chemical balance is maintained. Otherwise proper intumescent behavior may not happen.

Due to the wide range of applications and advantages of using intumescent paints as fire retardant coatings, there is substantial research on intumescent coatings, especially, involving the composition. Recently, nanocoating based intumescent coatings are now available.9 Literature exists on the methods for testing intumescent paints, using cone calorimeter,10,11 premixed flame12 and even solar furnace.13 The comparison of the intumescent paints is carried out mainly based on their behavior under different heat fluxes using cone calorimeter. Large amount of research work is reported on the modeling of the intumescent coatings behavior. Mesquita et al.14 considered intumesced char as a solid body and assumed one-dimensional conduction to determine the thermal conductivity of the char as a function of time and also used inverse heat conduction methodology to improve the thermal conductivity prediction. Stagg et al.15 shows that direct numerical simulation can also be used to estimate the thermal conductivity of the intumesced char. Zeverov et al.16 determines the thermo-physical property of the intumescent Paint as a function of temperature and has given a set of equations to predict the substrate’s behavior under premixed flame. Stagg et al.17 demonstrates the use of three dimensional thermal resistor networks to calculate the thermal conductivity and then compares with the analytical and experimental estimates of Al2O3.

Published research work on intumescent coatings is either performed in a closed furnace using ISO 864 curve’s temperature or with the help of cone calorimeter which have the capability of providing constant heat flux. But, accidental fires that occur in an open area like warships or any other structure that is exposed to hydrocarbon pool fires pose much harsher environment
to the intumescent coatings. Hence, there is a need to study the intumescent coatings in open pool fires. The present work concentrates on the behavior of the intumescent paints under different heat release rates using 0.5 m and 0.7 m open pool diesel fire. The present work demonstrates that the conclusions drawn on the basis of cone calorimeter experiments are not sufficient for real life application purpose. The paints need to be tested in real fire conditions. Temperature reduction ratio is used to determine the safe thickness for a given heat release rate of the heating source. The thermal conductivity of the paint is also calculated using the methodology given by Qiang et al.\textsuperscript{17} and the fire temperature distributions given by Sudheer and Prabhu.\textsuperscript{18}

**Materials and Methods**

**Experimental set-up and procedure**

Mass loss cone calorimeter (FTT make) is used as the controlled heat flux source as shown in Appendix Figure 1. It consists of the heating coil at the top, a sample holder and a load cell. The calibration of the heating coil is carried out with the help of a heat flux sensor (Schmidt-Boelter Heat Flux Sensor) at a distance of 60 mm from the bottom of the heating coil. Therefore, target plates are placed on a standard sample holder of the cone calorimeter above the ceramic blanket. Ceramic blanket of 25 cm thickness is filled in between the back side of the plate and the holder to avoid the heat loss from the back of the plate.

Appendix Figure 2 shows the schematic of the experimental set-up. Circular containers of 0.5 and 0.7 m made of mild steel are used for conducting open pool fire experiments. The containers are filled with 10-12 and 20-25 liters of diesel, respectively. Fire is initiated by pouring 10-20 ml of gasoline which acts as an igniter. Experiments are performed in the early hours of the day (5 pm-7 pm) so that the wind velocity is minimal. The experiments are conducted until steady state temperatures of the plate are achieved. The location of the coated Stainless Steel 310 plate is at \(Y/D=0.22\) from the fuel surface and at \(X/D=0.13\) as shown in Appendix Figure 2. Three thermocouples (k-type) are welded on the back side of the Stainless Steel 310 plate.

Experiments are conducted on the bare Stainless Steel 310 plates (\(d=2\) mm, 4 mm and 6 mm thickness) of 100 mm x 100 mm in cone calorimeter (50 kW/m\(^2\)) and 0.5 m open pool diesel fire. It should be noted that the Paint A has weak adhesive property compared to Paint B. For cone calorimeter experiments, a Stainless Steel 310 (2 mm) is coated with a dry film thickness (dfi) of 1.5 mm and 2.5 mm. Experiments are conducted at a heat flux of 50 kW/m\(^2\). Intumescence rate is captured using thermal infra-red camera (Thermoteknix VisIR 320-P) and digital camera (Canon PowerShot SX110 IS). For 0.5 m open pool fire experiments, three thicknesses of Stainless Steel 310 plates (2 mm, 4 mm and 6 mm) are coated with dry film thickness of 0.5 mm, 2.5 mm and 3.5 mm of paint A and paint B. The optimum thickness of paint B for a given heat release rate of heating source is arrived by conducting experiments both in 0.5 and 0.7 m open pool diesel fire.

**Figure 1. Temperature and energy absorbed by Stainless Steel 310 plates exposed in cone calorimeter and 0.5 m pool fire. SS310, stainless steel 310.**

\[\text{Page 2}\] [Fire Research 2019; 3:45]
Results and Discussion

Behavior of bare plate in cone calorimeter and open pool fire

Experiments are performed for different plate thicknesses (2 mm, 4 mm and 6 mm) with varying heat fluxes (35 kW/m², 50 kW/m² and 75 kW/m²) in cone calorimeter and also in open pool diesel fire. At any given instant, the temperature measured by three thermocouples are averaged and shown in Figure 1. Energy absorbed by the plate contributes to the rise in temperature of the plate. This is calculated using the heat equation shown below:

\[ \text{Energy absorbed by the plate} = (\rho V c_p) \frac{dT}{dt} \quad (1) \]

Variation of temperature and energy absorbed by the plates with time (for 2 mm, 4 mm and 6 mm) are shown in Figure 1. Temperature of the plate for pool fire keeps on fluctuating after reaching the maximum temperature. This is usually because of the wind which disturbs the steadiness of the fire/flame. The steady state temperature of the plates is around 770°C irrespective of the thickness of the plate in an open pool fire environment. However, the steady state temperatures are different for different thickness in cone calorimeter for a given heat flux (d=2, 4 and 6 mm). The temperature distribution suggests that the fire environment poses more severity on the bare plate compared to the cone calorimeter even at higher heat flux of 75 kW/m². Hence, it may be concluded that the results inferred from cone calorimeter may not be valid for open pool fire environment. It should be noted that the pool fire experiment didn’t last 20 minutes longer in case of d=4 mm and thus there is dip in the temperature.

Comparison of paint A and paint B in cone calorimeter

Influences of dry film thickness (1.5 mm and 2.5 mm) on the temperature, energy absorbed, mass loss rate and intumescent height of a stainless steel plate exposed to 50 kW/m² in a cone calorimeter is studied. Two paints considered in this study are paint A and paint B.

Figure 2 and Appendix Figure 3 show the transient temperature distribution of the stainless steel plate coated with a dry film thickness of 1.5 mm and 2.5 mm respectively. The performance of paint A and paint B appears to be almost same in terms of energy absorbed. However, from thermal shielding perspective, paint A shields slightly better in terms of temperature variation of the steel plate in comparison with paint B.

Figure 3 shows the mass loss fraction (mass of the paint at any instant/initial mass of the paint) of the intumescent paints. The mass loss fraction for the paint B is higher as compared to the paint A for both dry film thickness (1.5 mm and 2.5 mm). This shows that the initial thermal shielding contribution by the paint B may be more due to the evolution of the gases as compared to the paint A. However, in paint A, the contribution of gaseous evolution towards the cooling effect may be less predominant.

Appendix Figure 4 shows the intumescent thickness development for the paint A and B with an initial dry film thickness of 1.5 mm and 2.5 mm. The final intumescent height for the paint B is higher compared to that of paint A. One of the probable reasons for higher intumescence is the total volume of gaseous evolution. Porosity is directly proportional to the gases evolved during the decomposition. Although, the porosity and solid fraction of both the intumesced char is different, the effective thermal conductivity is close enough that intumesced char allows almost same heat transfer through it.

Comparison of paint A and paint B in a 0.5 m open pool diesel fire

In the previous section, comparison of paints in cone calorimeter environment is studied. Cone calorimeter is often preferred because of clean heating environment and a good control on the heat flux. Several differences between the environment in fire and cone calorimeter are as follows: i) varying heat flux with time in an open pool fire as opposed to constant heat flux in cone calorimeter; ii) sooty flame environment in pool fire compared to clean heating in cone calorimeter; iii) forced convective environment exists in pool fire (greater than 0.3 m pool fire) as opposed to natural convective environment in cone calorimeter. It may be concluded that the open pool fire environment is more hostile than the cone calorimeters (Figure 1). Hence, a better paint is to be accomplished on the basis of pool fire experiments rather than cone calorimeter experiments.

Figure 4 shows the live image of the experiment with 0.5 m open pool diesel fire. The image clearly shows the unsteady nature of the open pool fire. Due to the unsteady behavior of the flame, experiments are repeated more than twice and are averaged to get the final readings. Variations of temperature along with the energy absorbed by the Stainless Steel 310 plates (2 mm) with time (for different dry film thickness of the two paints) are
shown in the Appendix Figure 5.

Paint A performs similar to paint B for a dry film thickness of 0.5 and 1.5 mm. But with dry film thickness greater than 1.5 mm, the temperature profile of the plate is less predictable for Paint A. This is because the intumesced char formed by paint A falls off during the course of the experiment. The temperature distribution and the energy absorbed by paint B follow a smooth curve for different plate thickness and dry film thicknesses. For initial 4-8 minutes, the variation of the temperature for both the paints is same. But with the progression of time, paint A suddenly deviates from the smooth behavior. The reason behind this sudden change in the nature of the curve is the falling off of the swelled char from the plate leaving the plate unprotected. The peeling of the swelled char from the metal plate results in the disturbed energy absorption profile of the plate. Therefore, although the insulation property of paint A is good, due to its weak adhesive property, paint A fails in protecting the metal plate from high temperature exposure. On the other hand, the temperature profile and the energy absorbed distribution for Paint B follows a smooth behavior for Stainless Steel 310 plate thicknesses (2 mm, 4 mm and 6 mm) and different dry film thicknesses. The energy absorption rate is high for initial 1-2 minutes. However, with the passage of time, energy absorption rate decreases. This is because of the formation of the porous char due to high heat flux exposure. The adhesive strength of paint B upon swelling is better than Paint A.

Optimum thickness of paint B for 0.5 and 0.7 m diameter open pool diesel fire

The heat release rate for 0.5 m and 0.7 m open pool diesel fire is 0.207 MW and 0.502 MW.\(^{18}\) Figure 5A shows the condition of stainless steel plate coated with a dry film thickness of 1.5 mm of paint B. Paint B intumesces gets transformed into highly porous intumesced char in cone calorimeter (as presented in previous section). In cone calorimeter, a 1.5 mm dry film thickness of paint B expands around 18-20 times of its initial thickness. However, in an open pool fire, a 1.5 mm dry film thickness of paint B expands to only 10 times of its initial thickness (Figure 5A). Figure 5B shows the front view of the intumesced char on Stainless Steel 310 plate after the exposure to fire. The surface of the intumesced char is covered with soot from the diesel pool fire. This kind of participating behavior of the pool fire may be responsible for lesser intumescent height development. However, exact chemistry is not understood. SS310 plate can withstand a maximum temperature without thermal damage is 700°C.

Appendix Figure 6A shows the temperature variation for Stainless Steel 310 plates of different thicknesses (2 mm, 4 mm and 6 mm) coated with 4 different dry film thickness (0.5 mm, 1.5 mm, 2.5 mm and 3.5 mm) of paint B in 0.5 m open pool fire. Each experiment is repeated twice. There is improvement in the insulation capacity of the intumescent paint with the increase in dry film thickness, irrespective of the plate thickness. The temperature profiles with dry film thicknesses of 0.5 mm and 1.5 mm cross each other. However, there is a monotonic decrease in the temperature profile with the increase in the dry film thickness for thickness greater than or equal to 1.5 mm. There is a monotonic decrease in the energy absorbed by the plate with the increase in the dry film thickness of the paint. The temperature profile for 2.5 mm dry film thickness of the paint has lower slope compared to 0.5 mm and 1.5 mm paint thicknesses. 2.5 mm dry film thickness can hold the fire for more than 25 minutes for plate thickness of 2 mm. However,
after 25 min, the steel temperature reaches the 600°C mark. Similar behavior is observed for 4 mm thick Stainless Steel 310 plate. However, for 6 mm plate after 18 minutes, the temperature profile for 2.5 mm dry film thickness merges with temperature profile for 0.5 mm thickness. On the other hand, 3.5 mm dry film thickness for 6 mm plate appears to be better insulating thickness. Hence, maximum temperature attained by a Stainless Steel 310 plate with 3.5 mm dry film thickness decreases with the increase of Stainless Steel 310 plate thickness. This is because of the thermal inertia of the Stainless Steel 310 plate.

Appendix Figure 6B shows the energy absorption rate for Stainless Steel 310 plates of different thicknesses (2 mm, 4 mm and 6 mm) coated with 4 different dry film thickness (0.5 mm, 1.5 mm, 2.5 mm and 3.5 mm) of Paint B in a 0.5 m open pool fire. The energy absorbed by bare plate is the maximum. Energy absorbed by the SS 310 plate decreases with increase in the dry film thickness of paint B. Figure 6A shows the temperature variation with time and Figure 6B shows the energy absorption rate for 0.7 m open pool fire. The steady state temperature for Stainless Steel 310 bare plates is 827°C as against 777°C in case of 0.5 m pool fire. This variation in the steady state temperature is due to the difference in the heat release rates of the two pool fires. The temperature distribution for all the dry film thicknesses of paint B shows the expected behavior for initial 6-8 minutes. The temperature of the Stainless Steel 310 plate decreases with the increase in the dry film thickness of the paint B. Except for 1.5 mm dry film thickness of paint B, all other dry film thicknesses are showing monotonous behavior for 20 minutes. Thus, from the temperature distributions of Stainless Steel 310 plates in 0.7 m open pool fire, a 3.5 mm dry film thickness of paint B is showing better insulation property compared to the other dry film thicknesses. The difference in the slopes of the temperature distribution of Stainless Steel 310 plates for dry film thicknesses (0.5 mm, 1.5 mm, 2.5 mm and 3.5 mm) in a 0.5 m open pool fire is greater compared to 0.7 m open pool fire.

**Temperature reduction ratio**

The temperature reduction ratio (TRR) is defined as ratio of the difference in steady state temperature of bare plate and plate coated with paint to that of the bare plate. TRR is obtained for various combinations of plate thickness and paint thickness.

\[
\text{Temperature Reduction Ratio} = \frac{T_{\text{bare}} - T_{\text{paint}}}{T_{\text{bare}}} \tag{2}
\]

This parameter gives a clear indication of the effect of the paint thickness on the plate temperatures. Higher the TRR, better is the performance of the paint.

The TRR values obtained for Stainless Steel 310 plates in 0.5 m and 0.7 m pool fire are presented in Table 1. TRR is observed to be in the range of 0.1-0.4. In general, TRR is found to be increasing as the dry film thickness increases. 3.5 mm dry film thickness appears to be the best among other thicknesses covered in this study. The temperature reduction ratio is different for different thicknesses of Stainless Steel 310 plate for 3.5 mm dry film thickness. This is because, the temperature of the coated plate decreases with the increase in the thickness of the plate. This observation is in line with the findings made in Appendix Figure 6A.

**Prediction of transient temperature distributions of stainless steel 310 plate painted with paint B**

The prediction of the transient temperature distribution is essential in real life applications. This involves the estimation of effective thermal conductivity of char using one dimensional heat transfer model sug-
The governing heat equation for one-dimensional heat transfer is given as:

$$\frac{\partial^2 T(x,t)}{\partial x^2} - \frac{\partial T(x,t)}{\partial t} = 0$$  \hspace{1cm} (3)

where, $a_i = \frac{k_i}{\rho_i c_{pi}}$ is the thermal diffusivity, $k_i$ is the thermal conductivity of the insulation and $\rho_i c_{pi}$ is the volumetric specific heat of the insulation.

At the steel and the insulation interface, the boundary condition is given by:

$$-k_i \frac{\partial T_i(d_f, t)}{\partial x} = \frac{\rho_i c_{pi}}{A_i/V} \frac{\partial T(d_f, t)}{\partial t}$$  \hspace{1cm} (4)

where $C_{pi}$ is the volumetric specific heat of the steel; $A_i/V$ is the section factor, in which $A_i$ is the appropriate area of the fire insulation material per unit length, and $V$ is the volume of the steel per unit length; and $d_f$ is the thickness of the insulation. Dirichlet boundaries boundary condition at the fire-insulation interface is given as:

$$T(0,t) = T_f(t)$$  \hspace{1cm} (6)

where $T_f(t)$ and $T(0,t)$ are the fire temperature and the insulation temperature respectively. Qiang et al.\(^{17}\) demonstrated that Dirichlet boundary condition can be used and an equivalent thermal resistance can be calculated very easily and finally the thermal conductivity of the insulation.

Ignoring the heat absorbed by the insulation material, using the energy balance the steel temperature can be calculated by:

$$\Delta T_o = \frac{T_f(t) - T_{fo}}{R_{eq}}$$  \hspace{1cm} (7)

$R_{eq}$ can be calculated after rearranging the above equation as:

$$R_{eq} = \frac{T_f(t) - T_{fo}}{\Delta T_o} = \frac{A_i}{V} \rho_i c_{pi}$$  \hspace{1cm} (8)

Once the equivalent thermal resistance is known, the effective thermal conductivity can be easily calculated using the following relation:

$$k_{eff} = \frac{d_f}{R_{eq}}$$  \hspace{1cm} (9)

where $d_f$ is the initial dry film thickness of the intumescent paint applied on the substrate. The temperature at the fire and insulation interface is taken from Sudheer and Prabhu\(^{18}\) at an $X/D$ of 0.12 and $Y/D$ of 0.18-0.22 for both the 0.5 m and 0.7 m open diesel pool fire as 777°C and 827°C respectively. Using Eq. 8, the thermal resistance is calculated and from the thermal resistance profile an average value is chosen to determine the constant thermal conductivity of the insulation. Appendix Figures 7 and 8 show the comparison between the temperatures calculated using the thermal conductivity (Eq. 9) and the experimental data for 0.5 m and 0.7 m open pool fire respectively. This comparison is shown only for a Stainless Steel 310 plate of 6 mm thickness. The deviation in the measured temperatures of the plate and the temperature of the plate predicted using effective thermal conductivity is not more than 15%. The effective thermal conductivity is a function of paint thickness, substrate thickness and heat release rate (pool diameter).

Table 2 shows the thermal conductivity values for different Stainless Steel 310 plate for various dry film thicknesses of paint B in 0.5 m and 0.7 m open pool fires. It reveals that the thermal conductivity value is directly proportional to the thickness of the plate.

### Table 1. Temperature reduction ratio for a 0.5 and 0.7 m open pool diesel fire.

<table>
<thead>
<tr>
<th>Plate thickness (Stainless steel 310) (mm)</th>
<th>Dry film thickness (mm)</th>
<th>Temperature reduction ratio</th>
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<tr>
<td></td>
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<td>0.5 m pool</td>
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<tr>
<td>2</td>
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### Table 2. Effective thermal conductivity for paint B in 0.5 m and 0.7 m open pool fire.

<table>
<thead>
<tr>
<th>Stainless steel 310 (mm)</th>
<th>Open pool fire (mm)</th>
<th>Dry film thickness (mm)</th>
<th>Thermal conductivity (W/m·K)</th>
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<td>0.5 m pool</td>
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property; iii) evaluation of paint B in 0.5 m and 0.7 m pool fire experiments suggests that a dry film thickness of 3.5 mm is better compared to other thicknesses (0.5 mm, 1.5 mm and 2.5 mm) covered in this study; iv) estimated effective thermal conductivity of intumescent paint exposed to pool fire is suggested for a given pool fire diameter, dry film thickness and Stainless Steel 310 plate thickness. This thermal conductivity is able to predict the temperature variation of the plate within 15%.

References