



# Influence of titanium dioxide on the thermal insulation of waterborne intumescent fire protective paints to structural steel



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## ABSTRACT

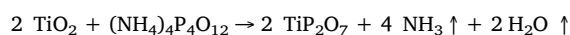
Titanium dioxide (TiO<sub>2</sub>) is an inevitable ingredient that influences the properties and performance of intumescent paints. The aim of this study is to investigate the effect of TiO<sub>2</sub> on the thermal insulation of waterborne intumescent paints. Five intumescent formulae with different weight ratios of TiO<sub>2</sub> to ammonium polyphosphate (APP), including paint without TiO<sub>2</sub> as a control sample was prepared. Thermal properties and thermal insulation performance of intumescent paints were measured by thermogravimetric analysis (TGA) and customized programmable furnace, respectively. The composition of char was analyzed by using X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, and inductively coupled plasma-optical emission spectroscopy (ICP-OES). Results showed that ratio of both ingredients (TiO<sub>2</sub>:APP) play a critical role on the performance of intumescent paints. It is suggested that ratio of slightly excess stoichiometric atomic ratio of titanium to phosphorus (Ti:P) exhibits better thermal insulation of intumescent paints.

## 1. Introduction

Intumescent paints are an effective passive fire protection materials widely used in steel construction. Although structural steel is inherently nonflammable but it loses its strength when exposed to fire of higher temperature and also unprotected steel heats up very rapidly resulting in premature collapse of building structures. This can be prevented or extended by structure that is installed with passive fire protection materials [1]. Intumescent paints are reactive coatings that react to heat by swelling in a controlled manner to many times its original thickness to produce a carbonaceous char, which acts as an insulating layer to protect the steel. Both solvent and water based intumescent paints are most commonly used to protect the structural steel components (Beams and/or Columns) in commercial buildings against cellulose fire of which fuel is cellulosic nature such as paper, wood, and fabrics [2]. Now-a-days, increasingly stringent regulations and legislation concerning organic solvent emissions has meant greater use of water based intumescent products.

The properties and performance of intumescent paints can be varied within limits by the choice of raw materials and method of applications [3]. The classical intumescent composition contains raw materials such as an acid source, a char source, the blowing agents, binders, pigments and specialty additives for easy processing, adhesion promotion, and rheological modification. For every ingredient plays an important role

on the performance of intumescent paints. It has been reported that at high temperature titanium dioxide (TiO<sub>2</sub>) reacts with ammonium polyphosphate (APP) or its degradation products to form titanium pyrophosphate (TiP<sub>2</sub>O<sub>7</sub>), which is thermally stable and decreases the radiative heat transfer from the surface thereby improving the heat barrier properties [4,5].



Titanium dioxide is most commonly used as a pigment in intumescent paints. It is chemically inert at room temperature, non-toxic and usually available in both rutile and anatase grades. It can deliver the opacity, whiteness and brightness to the intumescent paints. It can also promote the photo-degradation of most organic paints that can be overcome by using a pigmentary form of TiO<sub>2</sub> where the surface is coated with silica and alumina in various proportions [6]. The crystalline phase-II APP (chain length > 1000, less water soluble and high thermal stability) is a polymeric structure of the ammonium salt of polyphosphoric acid, which is widely used in intumescent paints for the fire protection of structural steel. The fire protective efficiency of APP is generally attributed to the increase of char formation through a condensed phase mechanism [7].

There are few reports available on the effect of TiO<sub>2</sub> on the fire protective behavior of intumescent coatings. Duquesne et al. have studied the influence of TiO<sub>2</sub> and mineral fiber on the fire protection of

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intumescent coatings by cone calorimeter using  $35 \text{ kW/m}^2$  radiant heat flux. It was found that coating with atomic ratio of titanium to phosphorus (Ti/P) of 0.5 showed better fire protection performance [8]. Another study reported 4% by weight  $\text{TiO}_2$  was optimum for better thermal insulation properties of amine cured epoxy intumescent coatings using Bunsen burner test [9]. Li et al. investigated that rutile  $\text{TiO}_2$  offered better fire retardant performance of wooden panel than that of anatase grade in waterborne intumescent coatings using Bunsen burner test [10]. However, none of these studies have reported the effect of  $\text{TiO}_2$  on the performance of intumescent paints at cellulosic fire heating conditions where the temperature reaches around  $1000^\circ\text{C}$ . Moreover, the present study is based on the composition with different weight ratios of  $\text{TiO}_2$  to APP, whereas the previous studies have shown the atomic ratios of Ti to P and varying the different concentrations of  $\text{TiO}_2$  and also using different parts per hundred of  $\text{TiO}_2$  by weight. Further, it was also observed that the different ratio of  $\text{TiO}_2$  to APP have been employed for commercial intumescent guideline formula that was received from different raw material suppliers.

This investigation reports the optimization of  $\text{TiO}_2$  to obtain the desired performance of waterborne intumescent paint, which gives better thermal insulation properties and also uniform char formation. The effect of different weight ratios of  $\text{TiO}_2$  to APP on the thermal decomposition, thermal insulation properties and char analysis was investigated. In this case, thermogravimetric analysis (TGA), customized programmable high temperature furnace, X-ray diffraction (XRD), attenuated total reflection mode of Fourier transform infrared (ATR-FTIR) spectroscopy and inductively coupled plasma-optical emission spectroscopy (ICP-OES) were used to characterize the properties of intumescent paints and their char and correlate them with atomic ratio of titanium to phosphorus and/or the weight ratio between  $\text{TiO}_2$  and APP. Importantly, all these properties of intumescent paints have been compared with those of the control intumescent paint, that is, prepared without  $\text{TiO}_2$  and also with unprotected or bare steel plate.

## 2. Experimental

### 2.1. Materials

Raw materials such as vinyl acetate dispersion, ammonium polyphosphate (molar mass =  $97 \text{ g/mol}$  and phosphorus 32%), pentaerythritol, melamine, and rutile titanium dioxide (99% pure, molar mass =  $80 \text{ g/mol}$  and titanium 48%) were used as binder, an acid source, a char source, the blowing agent and pigment in paint formulation that were obtained from Visen Industries Ltd (India), Clariant (Switzerland), Asian Paints Penta division (India), JLS (China), and DuPont (India), respectively. Other additives, dispersing agent, rheology modifier, defoamer, etc. were supplied by different commercial sources. All raw materials were used as received.

### 2.2. Preparation of paints and test specimen

The formulation shown in Table 1 was used to prepare the

**Table 1**  
Chemical composition of intumescent paints.

S.No.	Raw Materials	NT	TA0.3	TA0.5	TA1	TA2
1	Water	20.00	20.00	20.00	20.00	20.00
2	$\text{TiO}_2$	–	7.50	12.70	20.63	30.00
3	APP	33.00	28.50	25.38	20.63	15.00
4	Pentaerythritol	11.00	9.50	8.46	6.87	5.00
5	Melamine	11.00	9.50	8.46	6.87	5.00
6	Additives	5.00	5.00	5.00	5.00	5.00
7	Binder	20.00	20.00	20.00	20.00	20.00
Total		100.00	100.00	100.00	100.00	100.00

NT – Nil  $\text{TiO}_2$ ; TA – ratio between  $\text{TiO}_2$  and APP (ammonium polyphosphate).

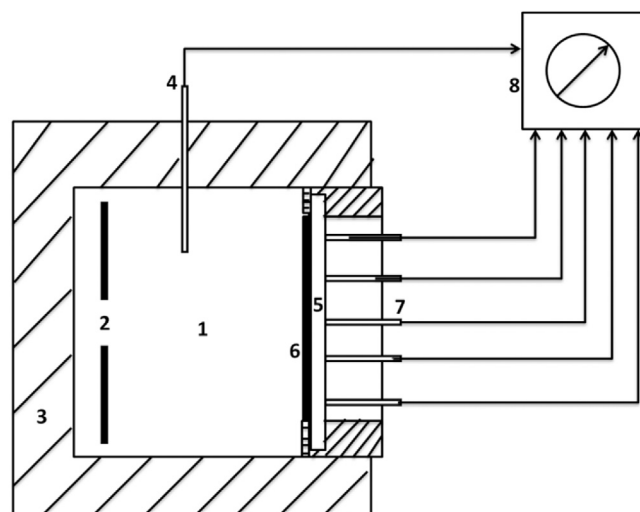
intumescent paints with different ratios of  $\text{TiO}_2$  and APP. The prepared paint samples were designated as NT and TA (0.3, 0.5, 1 and 2) for compositions without  $\text{TiO}_2$  (control sample) and ratio by mass of  $\text{TiO}_2$  to APP, respectively. The waterborne intumescent paint was prepared in a beaker by mixing all ingredients using a high speed disperser. The order of addition of raw materials were same as shown in Table 1. A sufficient grinding time was allowed for a proper mixing and dispersion of ingredients. The known amount of intumescent paint was directly applied to the steel plate using a paint brush in a single coat application ( $500 \pm 50 \mu\text{m}$  dry film thickness). The coated plate was then allowed to air dry for 7 days for complete drying of paint.

### 2.3. Characterizations

The thermal properties were measured by Thermogravimetric analysis (TGA), TGA1 STAR system, Mettler Toledo, Switzerland under nitrogen or air atmospheres (flow rate of  $50 \text{ mL/min}$ ) at a heating rate of  $20^\circ\text{C/min}$  over the temperature range of  $30\text{--}1000^\circ\text{C}$ . Typical sample weights of  $3\text{--}5 \text{ mg}$  were employed.

The electrically heated programmable (rate of temperature rise is corresponding to the standard cellulosic fire curve as per ISO 834) bench scale furnace was used to measure the thermal insulation of intumescent paints. Fig. 1 shows the schematic diagram of furnace and assembled carbon steel plate ( $360 \times 360 \times 5 \text{ mm}$ ), which was sand blasted on one side (paint coated side) with a profile of approximately  $70 \mu\text{m}$ . The furnace was constructed by in-house facility and the necessary components were procured from local sources. The paint coated area of steel plate ( $250 \times 250 \text{ mm}$ ) and the temperature gradient measured points (location of thermocouples) at the backside of plate are shown in Fig. 2(a) and (b). Temperature of both furnace and plate was measured by using K-type thermocouples. The unexposed areas of steel plate (sides and edges) were protected by covering with heat resistant ceramic wool to prevent the heat loss. The temperature curve as a function of time was recorded until the backside mean temperature reached to  $500^\circ\text{C}$  as a critical or limiting temperature of steel plate. The thermal insulation time of three specimens per sample was measured and their average values are reported.

X-ray diffraction (XRD) was carried out by using X'Pert Pro Panalytical diffractometer, The Netherlands with  $2\theta$  angle range of  $4^\circ\text{--}60^\circ$ . An accelerating voltage of  $40 \text{ kV}$  and a current of  $30 \text{ mA}$  were applied using Ni-filtered  $\text{CuK}\alpha$  radiation. The acquisition parameters, step size of  $0.02^\circ$  and scan step time of  $2 \text{ s}$  were followed. Attenuated total reflection mode of Fourier transform infrared (ATR-FTIR) spectro-



**Fig. 1.** Schematic diagram of furnace. 1–furnace chamber; 2–heating coils; 3–heat resistant ceramic insulation; 4–furnace thermocouple; 5–steel plate; 6–coated intumescent paint; 7–plate thermocouples; 8–control unit.

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