

Investigation of Fire-Retardant Performance of Locally Formulated Intumescent Coatings Without Ammonium Polyphosphate on Engineering Materials.

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Abstract: This paper focuses on investigating the efficiency and evaluation of intumescent coating produced using ammonium phosphate, melamine, and pentaerythritol as core components and that produced without ammonium phosphate. Intumescent coatings provide passive fire protection by swelling when exposed to heat, forming an insulating char layer that protects underlying materials. The study involved formulating the coating, conducting quality tests such as pH, spreading rate, drying time, and stability, and performing a fire-resistance test. The quality test results showed that the coating met industry standards, though the drying time exceeded the ideal range. The fire-resistance test demonstrated the coating's ability to provide effective fire protection for 30 minutes. However, the absence of ammonium phosphate led to reduced fire-resistance duration as compared with coatings produced with ammonium phosphate. Overall, the coating showed potential for fire protection applications.

Keywords: efficiency; fire-retardant; intumescent; engineering material

I. INTRODUCTION

Intumescence is the swelling of a material in response to heat, usually resulting in the formation of a multi-cellular, carbonaceous, or ceramic layer (Jimenez et al., 2006). This layer serves as a thermal barrier, effectively shielding the substrate from sudden temperature increases and preserving its structural integrity (Mariappan, 2016; Horrocks & Price, 2008). People and the structures they erect are seriously threatened by fire. To stop its effects on them, newer techniques and materials are constantly being developed. A more recent kind of passive fireproofing coating is the intumescent coating.

It is typically applied as a thin film and expands many times its original thickness to create an insulation char that serves as a barrier between the fire and the structural member (Puri & Khanna, 2016). The char completely shields the substrate from an unnecessarily high temperature and oxygen intake, thereby hindering the fire from burning further (Omrane et al., 2007; Władyka-Przybylak & Kozłowski, 1999).

Moreover, effective intumescent coatings depend on the careful selection of carbonizing, foam-producing,

and dehydrating materials, among other essential components (Sami et al., 2017; Kozłowski & Władyka-Przybylak, 2008). The component parts of intumescent coatings are an acid source that generates an acid catalyst and a gas source that acts as a blowing agent (Li et al., 2022). It also consists of a carbonization agent, also known as a char-forming agent (Riva et al., 2003).

One common phosphorus-containing flame-retardant is ammonium phosphate (Hassan et al., 2015). It has many benefits, including low toxicity and smoke production—two qualities common to flame-retardants based on phosphorus (Zhang et al., 2019). Furthermore, ammonium phosphate is inexpensive, environmentally benign, and has high flame retardancy. Through intumescence, ammonium phosphate can act as a flame-retardant in the condensed or polymer phase. (Lim et al., 2016).

The conflagration of buildings has a significant negative influence on the economy and society each year and

results in many fatalities worldwide (Lataille, 2002). Even though building fires are thought to occur infrequently, a fire can have disastrous effects; for this reason, in situations where fire could be an issue such as high-rise structures, protective means that could minimize such fire incidents such as intumescent coatings are developed (Buchanan & Abu, 2016).

Improper storage of flammable substances such as petrol and gas, largely due to frequent power outages in Nigeria, and the use of sub-standard building materials, among many other causes has led to the existing trend of fire outbreaks in buildings. Thus, among all the other factors in the design process of a fire-safe structure, structural integrity and stability constitute a fundamental component of the fire safety strategy (Maluk et al., 2017).

One of the biggest avoidable tragedies in contemporary society is fire loss. Annually, fire results in significant loss of life and property. Research on fire-retardants such as intumescent coatings is therefore given a lot of attention to stop the loss of lives and property (Jimenez et al., 2006; Kirshenbaum, 2005).

Despite the significant progress in the development of intumescent coatings, several challenges remain unaddressed. These include optimizing the formulation of intumescent coatings using locally available raw materials. Addressing these challenges requires a comprehensive understanding of the underlying mechanisms governing the fire-retardant properties of intumescent coatings and their interaction with different substrates such as steel, wood, and concrete (Yao et al., 2019; Shan-Shan et al., 2019).

The fire-retardant efficiency of intumescent coatings without ammonium polyphosphate on engineering materials is lacking in literature. This knowledge gap is what this paper tends to address by using adjustable formulation ratios of the raw materials sourced locally to produce a reliable fire-retardant coating that can protect lives and property at reduced cost.

It is the purpose of this paper to produce intumescent coatings of adjustable ratios that could act as fire retardants on engineering materials to prevent any serious loss that might arise in case of fire out-break.

1. Materials and Methods

2. Materials

Materials and equipment used for the study include Water (solvent), Acrylic, Titanium dioxide, Cellulose ether, Calgon, Defoamer, Butyl alcohol, Texanol, Ammonia, Melamine, Pentaerythritol, coating mixing vessel, Manual mixer, Analytical balance, Measuring cylinder, paint brush, Personal Protective Equipment (gloves, goggles, nose masks, boots and coats), Thermometer, pH meter.

II. METHODS

The methods outlined below were used to formulate, prepare, and test the intumescent coatings.

2.1 Coating Formulation

Table 1 displayed the raw materials and measurements that were used to produce the intumescent coatings.

Table 1: Raw materials and quantities

S/N	Raw Materials	Amount
1.	Water	280ml
2.	Acrylic	140g
3.	Titanium dioxide	60g
4.	Cellulose ether	3g
5.	Calgon	1.6g
6.	Defoamer	3g
7.	Butyl alcohol	10ml
8.	Texanol	10ml
9.	Ammonia	10ml
10.	Melamine	360g
11.	Pentaerythritol	60g

1. Preparation Procedure

- Titanium dioxide was dissolved in water and calgon was added to help disperse it quickly in the coating mixing vessel.
- Acrylic was added and stirred continuously.
- Defoamer was added and stirred until the lather disappeared.
- Cellulose ether was dissolved in small one litre of water and added to the coating mixing vessel. It was stirred until the desired viscosity was obtained.
- Butyl alcohol was added and stirred.
- vi. Texanol was added and stirred.
- Melamine was added and stirred.

- Pentaerythritol was added and stirred.
- Ammonia was added and stirred until the mixture became homogeneous

2. Quality Tests

This is to ensure the coating meets established standards, market specifications, and consumer needs. The parameters tested include pH, colour, temperature stability, spreading rate, surface, drying time and flakiness.

Colour: The coating was applied to a clean, dry surface, and its colour was observed.

Flakiness: The surface of the dried coating was checked for flakiness and staining possibilities

pH Test: Coating of 20 grams was measured into a 100 ml beaker, and 50 ml of distilled water was added. The mixture was stirred, and the pH was measured.

Temperature Stability at 50°C: Fifty (50) grams of coating was heated to 50°C in a crucible and allowed to cool. Observations were made for colour separation, floating, or cracking.

Spreading Rate: Ten (10) grams of coating was applied with a brush to a clean, dry surface, and the area covered was recorded.

Surface Drying Time: The time taken for the applied coating to lose its moisture content was observed and recorded.

3. Fire-Resistance Test

This test shows how well the prepared coating will resist fire. Materials used include prepared coating, Oxy-acetylene flame, Thermocouple wires, Temperature control meter, mild steel substrate sample.

4. Experimental Set-Up

The substrate is coated with the intumescent coating on one side. Thermocouple will be connected to the uncoated side of the substrate as shown in Figure 1 below. The flame is focused on the coated surface of the substrate at 25mm to 50mm.

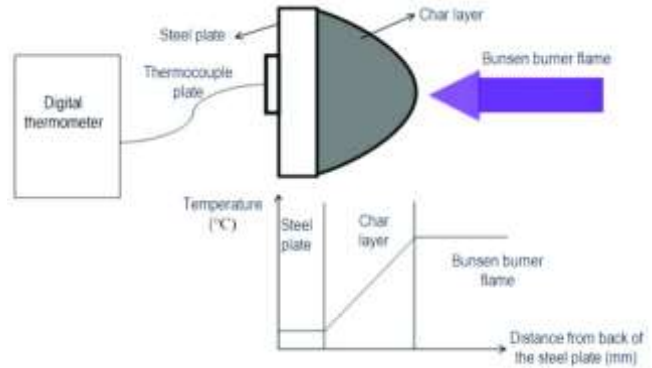


Figure 1: Experimental setup for fire-resistance test - Source: Torbjorn, (2019).

Test Procedure

The oxyacetylene flame was applied on the coated surface of the substrate for thirty minutes. The temperature increase for every five minutes was recorded.

III. RESULTS AND DISCUSSION

1. Results

Table 2: Results of the quality tests conducted on the produced intumescent coating.

Parameters	Test	Standard
Colour	White	
pH	7.5	6.5 - 7.5
Temperature stability	Stable at 50°C	Stable at 50°C
Spreading rate (coverage)	10g of coating covered 2.09m ²	10g per 0.16m ²
Surface drying time	114 minutes	30 - 90 minutes
Flakiness.	No flakiness	No flakiness

Blowing agents, such as melamine, released gases upon decomposition, causing the intumescent coating to swell and form an expanded foam structure. This foam layer acts as an insulator and barrier to heat and fire. In their multi-scale experimental approach, Jimenez et al., (2006) discussed the role of blowing agents in intumescent coatings. They emphasized that the synergy between the blowing agent and other components is crucial for achieving optimal expansion and insulation properties.

Binders and resins hold the intumescent ingredients together and ensure that the coating adheres well to the substrate. Common binders include epoxy, acrylic, and polyurethane resins, which also contributed to the overall durability and flexibility of the coating. According to Strassburger et al. (2022), different water-based resins significantly influenced the performance of intumescent coatings. They compared various resins and highlighted their impact on the film formation and adhesion properties of the coatings, which are essential for effective fire protection.

Additives such as pigments, fillers, and stabilizers are incorporated to enhance the performance characteristics of intumescent coatings. These may include UV stabilizers, plasticizers, and smoke suppressants to improve durability, flexibility, and reduce smoke generation during a fire. Zhang et al. (2019) explored the effects of different smoke suppressants in combination with ammonium phosphate on enhancing the flame retardancy and smoke suppression of vinyl ester resin systems. This study demonstrates how tailored additives can enhance the overall fire performance of intumescent coatings.

The effectiveness of intumescent coating is derived from the synergistic interaction of the afore-mentioned components. Upon exposure to heat, the acid source (e.g., ammonium phosphate) decomposes to form phosphoric acid, which catalyzes the dehydration of the carbon source (e.g., pentaerythritol) to create a char. Concurrently, the blowing agent (e.g., melamine) releases gases that cause the char to expand and form an insulating barrier. The binder ensures that the coating adheres well and remains intact during the intumescent reaction.

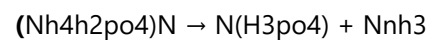
1. Chemical Reactions In Intumescent Coatings

The primary components of intumescent systems are typically an acid source, a carbon source, and a blowing agent. These components undergo specific chemical reactions under heat to produce the intumescent char. The following is a breakdown as seen in literature (Hassan et al., 2015; Lim et al., 2016; Horrocks and Price, 2008).

Ammonium Phosphate (AP) Reaction: Ammonium Phosphate acts as an acid source and is a common ingredient in intumescent formulations. It decomposes upon heating to release phosphoric acid, which then

catalyzes the dehydration of the carbon source (typically a polyol or a carbohydrate) to form a char layer. Hassan et al. (2015) discussed the use of ammonium phosphate in improving the flame retardancy and thermal stability of polymer composites. Ammonium Phosphate is emphasized as a crucial component in creating a robust and insulating char layer in intumescent systems, enhancing the fire resistance of the materials it is incorporated into.

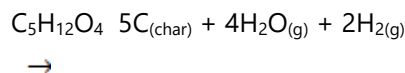
Upon heating, ammonium Phosphate decomposes to produce phosphoric acid and ammonia. Phosphoric acid acts as a catalyst for the dehydration of the carbon source. The chemical reaction is shown below:



Lim et al. (2016) detailed the role of ammonium phosphate in intumescent systems, highlighting its decomposition to phosphoric acid, which catalyzes the charring of the carbon source and promotes the formation of a stable protective layer.

Carbon Source - Pentaerythritol Reaction:

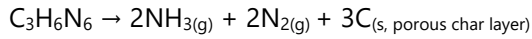
The carbon source in intumescent coating is responsible for forming the char structure upon thermal decomposition. Common char formers include pentaerythritol, dipentaerythritol, or starch. These substances dehydrate and carbonize to produce the protective char layer that insulates the substrate. Horrocks and Price (2008) provide a detailed account of how carbon sources function in intumescent coatings. They highlighted that the choice of carbon source significantly affects the intumescent process and the quality of the char produced. The dehydrating action of phosphoric acid on pentaerythritol leads to the formation of a carbonaceous char. This process involves complex reactions where pentaerythritol loses water molecules and forms a cross-linked char structure.



Jimenez et al., (2006) discussed the multi-scale approach in analyzing the formation of char in intumescent coatings, where pentaerythritol plays a critical role in generating a robust and thermally insulating char layer.

Melamine Reaction: Melamine decomposes upon heating to release non-flammable gases like nitrogen

and ammonia, which cause the intumescent mixture to foam and expand. This expansion forms a thick, porous char layer. The chemical reaction is shown below:



Horrocks and Price (2008) illustrated the role of melamine in intumescent systems, emphasizing its function in gas release and expansion, which enhances the thermal insulation properties of the char layer.

3. Fire Resistance Test

Table 3: Result of the Fire Resistance Test of the coating

Time (Mins)	Temperature (°C)
0	30
5	56
10	95
15	180
20	257
25	342
30	362

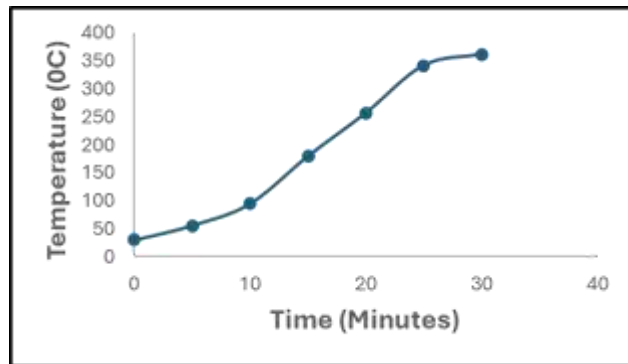


Figure 2: Fire Resistance Curve of the Intumescent Coating

Figure 2 shows the fire resistance graph of Temperature against Time. The fire resistance test showed that the coating can withstand significant temperature increases over 30 minutes, forming an insulating barrier. Figure indicates that the fire-rating of the coating is but for 30 minutes. This is due to the absence of ammonium phosphate which was not added to the final formulation used to produce the coating in this work. The result, however, showed that the absence of ammonium phosphate in the coating formulation caused a significant decrease in the fire rating of the coating. The

overall process involves the coordinated action of these components: the acid source catalyzes the dehydration of the carbon source while the blowing agent released gases to expand the char. The result is an expanded, insulating char that protects the underlying material. In their review, Puri and Khanna described the interactions between these components in forming the intumescent char. They highlighted how the acid source, carbon source, and blowing agent work in tandem to create a stable and expanded protective layer (Puri & Khanna, 2016). The synthesis of the interactions using Ammonium Phosphate (AP), pentaerythritol, and melamine as a typical intumescent system is well documented (Farzaneh et al., 2019). The char expansion was due to gas released from melamine, combined with the catalytic action of phosphoric acid on pentaerythritol. This results in an expanded, thermally insulating layer that protects the substrate from heat and fire (Ayman et al., 2017). The quality tests confirmed that the coating performs well in terms of pH balance, temperature stability, and flakiness. The coating also possesses good coverage in terms of spreading rate which makes the coating cost effective. This research work noted that the drying time of the coating produced exceeded the standard range by about 14 minutes.

IV. CONCLUSION

The research successfully produced intumescent coating without ammonium phosphate, and the coating underwent quality, and fire-resistance tests. The results from the quality tests showed the coating met industry standards in several key areas, including color, pH, temperature stability, spreading rate, and lack of flakiness. However, its drying time exceeded the standard range, indicating a potential area for improvement. The fire-resistance test confirmed that the coating provides significant protection against fire by forming an insulating char layer, though the coating's fire resistance was effective for a limited duration of 30 minutes. The absence of additives, like ammonium phosphate, contributed to the shorter fire protection time. Nevertheless, the overall performance of the coating shows promises for use in fire protection despite the absence of ammonium phosphate.

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